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OPERATION OF THE GOULDEN LARGE-
SAMPLE EXTRACTOR (GLSE) (LASALLE
SCIENTIFIC INC., VERSION GLSE-95)

D.H.J. Anthony and T. Nagami

RAB-TN-93-32

**OPERATION OF THE GOULDEN LARGE-SAMPLE EXTRACTOR (GLSE)
(LASALLE SCIENTIFIC INC., VERSION GLSE-95)**

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MANAGEMENT PERSPECTIVE

The Goulden Large-Sample Extractor (GLSE) was developed within the Analytical Chemistry Research Project (ACRP) of the Research and Applications Branch (RAB). It provides a means by which, through preconcentration by continuous-flow solvent extraction, detection limits for a comprehensive class of organic contaminants ("Priority Substances") could be lowered to the point that meaningful statements could be made about their distributions in freshwater systems. The technology was originally developed for use by the Water Quality Branch/Ontario Region in the Niagara River and Great Lakes Toxics Management Programs (NRTMP, GLTMP).

The GLSE technique has since received considerable interest on the part of other groups responsible for water quality monitoring (eg. the US EPA and the US Geological Survey) who share similar objectives in the ultratrace monitoring of organic contaminants in freshwater.

Because of early publications and wide-spread dissemination of information on the technique, it could not be patented. In order to assist interested groups in incorporation of the technology in their monitoring programs, the authors have set up an informal relationship to manufacture and commercialize the most "popular" GLSE prototype.

This report describes the operation of this GLSE prototype (GLSE-95) and is intended as a operating manual for LaSalle Scientific Inc. to provide along with the equipment.

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WAIVER

Her Majesty, as represented by the Minister of the Environment, and Environment Canada, the National Water Research Institute, the Research and Applications Branch, the Analytical Chemistry Research Project and the authors make no guarantees as to the performance of the GLSE-95 manufactured by LaSalle Scientific Inc. (Guelph, Ontario) in any application.

DISCLAIMER

Her Majesty, as represented by the Minister of the Environment, and Environment Canada, the National Water Research Institute, the Research and Applications Branch, the Analytical Chemistry Research Project and the authors assume no responsibility for damages or consequences of any kind resulting from the use of the GLSE-95 manufactured by LaSalle Scientific Inc. (Guelph, Ontario) in any application including, but not limited to, the proper disposal of extraction effluent.

The use of manufacturer's equipment or trade names does not constitute endorsement or certification by Her Majesty, as represented by the Minister of the Environment or by Environment Canada, the National Water Research Institute, the Research and Applications Branch, the Analytical Chemistry Research Project or by the authors for use in any application.

ABSTRACT

This report describes operation of the LaSalle Scientific Inc. version of the Goulden Large-Sample Extractor GLSE-95 prototype and the minimal set-up work involved.

This material serves only as an introductory manual for getting started in the application of GLSE technology to particular water matrices. References to more comprehensive descriptions of the technique are provided.

INTRODUCTION

The Goulden Large-Sample Extractor (GLSE) was designed to provide a convenient means by which a certain class of organic contaminants in large samples (50-200 L) of water could be conveniently concentrated to measurable levels for environmental analytical purposes (1-11). When incorporated in an appropriate ultratrace analytical method, the technique can provide data at the parts-per-trillion (ppt) and parts-per-quadrillion (ppq) levels necessary for sensitive monitoring of contaminant distributions and trends in environmental freshwater systems.

The GLSE is a continuous-flow solvent extraction apparatus designed on the mixer/settler principle and optimized to perform an efficient single-stage extraction of contaminants having low solubility (hydrophobic) in water. Many of these contaminants are included in "Priority Substances" lists of Canadian and U.S. governments (US EPA, CEPA, MISA). With this apparatus, continuous compensation is made for solvent loss due to its solubility in water, for continuous pre-heating of the sample to 20°C and for the continuous introduction of surrogate standards for the purpose of evaluating extraction efficiency.

The GLSE-95 apparatus is manufactured and marketed by LaSalle Scientific Inc. as a package for those users who prefer not to assemble the apparatus "from scratch". The apparatus is modelled after the GLSE-95 prototype originally described by Goulden and Anthony (1). An intentional effort has been made to use components, aside from the custom-made glassware, which are easily sourced, rather than to develop and market the apparatus as a user-non-serviceable "black box".

The principal components of the apparatus and their orientation are shown in Figs. 1-4. The parts list is given in Appendix A.

An extensive list of documented applications of the GLSE-95 prototype is provided (Appendix B). The user is advised to consult this information before using the apparatus in a particular application. For applications outside those documented, the user is obliged to perform the necessary research and validation work before any data generated is used.

PRE-INSTALLATION

Pre-installation requirements for the GLSE are minimal.

The following provisions should be made :

1. Due to the hazardous nature of the solvent used (1,2-dichloromethane (DCM), alias "methylene chloride"), adequate ventilation is important. A fume-hood or fume-cabinet would be the ideal location but any well-ventilated laboratory benchspace is satisfactory, particularly if located near a fume-hood or fume cabinet.

Personnel using or working in the vicinity of the extractor should be familiarized with the hazardous properties of the solvent thru the appropriate MSDS.

Government (municipal, provincial, federal) regulations or guidelines applying to hazardous waste discharge should be consulted to determine how extraction effluent will be disposed of (the solubility of the extractant (DCM) in water is 1.6%w/w, 1.3%v/v).

2. Access (2-3 ft.) to a standard 115 VAC/15 A utility outlet.
3. Access (2-3 ft.) to a sink, benchtop or floor drain.

The extractor cabinet, accessories and workspace will occupy about 5-6 ft. of bench length.

SET-UP

Bold terms indicate terms defined in Figures.

The GLSE-95 is supplied partially assembled in a carrying-case and only minimal assembly is required for those components which might cause damage to the glassware or become dislodged during shipping.

Before proceeding with set-up, ensure that the components are free of any debris from packing material and that the surfaces of glass "pick-up" and delivery tubes are rinsed free (eg. methanol, dichloromethane (DCM)) of any glue residue from tape used to hold the pieces during shipment.

- using a screw-driver, loosen the mounting straps holding the **separator trap** and re-orient the **separator trap** so that the **extractor drain** is located in the **separator trap inlet** as shown in **Fig.5**. Re-tighten the mounting straps and connect the **separator trap outlet** to a drain using Tygon tubing. Ensure that this drain tube is "free-flowing" and not constricted or of such length so as to develop "plugs" of water in the drain tubing. This may cause overflowing of the **separator trap**.

- if the **sample pump outlet line** has not been connected to the **heater tube** install it on the **heater tube inlet** at this time. Loosen the heater tube mounting straps using a screw-driver. Expand the end of the Teflon-lined polyethylene (PE) tubing slightly, (heating the tubing end in boiling water will soften it temporarily), using a twisting motion, with the tool provided

or a 10 mL transfer pipette. Friction-fit the tube on the inlet to a sufficient length ($3/8$ to $1/2$ inches) and seal the fitting with a hose clamp (provided) (Fig.6).

- if the **sample pump output line** was connected on shipment, loosen the mounting straps holding the **accessory tube (heater tube)** using a screw-driver.
- install the Teflon "**drip-tip**" on the tube's outlet and re-orient the tube so that the outlet is centered in the **extractor inlet** as shown in (Fig.6). Re-tighten the mounting straps.
- fit the two Teflon **spacers** on the **silica immersion heater**, so that when installed in the **heater tube**, one is located near the bottom (~2" from bottom) and one near the top of the **heater** (Fig.6). Clamp the **heater** in place, with the "micro-clamp" fitted over the plastic end-cap of the heater, to the smaller mounting post.
- if not already fitted, press a stainless steel insert (Fig.6) (provided) into the other end of the Teflon-lined PE tubing after it has been cut (scalpel, or X-acto knife) to a length sufficient to reach the **sample pump** without excessive stress.
- using a long "needle-nose" tweezers, lower the Teflon **scrubber column base plate** into the **column form** and rest it on the restricted end of the form (Fig 7). Install the solvent "**make-up**" **delivery tube** in the **column form** so that it rests close to the **base plate** and tape the other end of the delivery tube to the outer surface of the "**scrubber column**" (Fig.7). (If the Teflon solvent "**make-up**" line has not already been threaded through the **delivery**

tube, do this before installing the delivery tube; the end of the Teflon tubing should extend $\sim 1/4$ " beyond the end of the delivery tube) (Fig.8).

- hold the Teflon restrainer in place so that the perforations are centered over the inlet of the extractor drain, and pour the Teflon "Raschig Rings" packing into the scrubber column form until the level is ~ 1 " above the restrainer. The column packing will hold the restrainer in place (Fig.7).

The scrubber column functions adequately with the packing loosely-packed. Do not, at any time, exert downward pressure on the column as this will drive the base plate through the restriction allowing packing to fall into the extractor. If replacing or trying to orient the solvent "make-up" delivery tube, use a "stirring" motion with little downward pressure.

- if not already installed, thread the intake portion of the Teflon solvent delivery line through the "solvent make-up" pick-up tube so that it protrudes $\sim 1/4$ " from the end of the tube (Fig.8).
- similarly, install the surrogate standards intake line in its pick-up tube (Fig.9) and the delivery line in its inlet on the heater tube (Fig.6).
- install the thermometer provided in the thermometer port of the heater tube (Fig.6).
- fix the impeller in the stirrer so that the impeller will be roughly in the recommended position when the stirrer is mounted (Fig.10). Fit the impeller blades through the stirrer port of the extractor and lower the stirrer until the impeller is located, vertically, approximately at the position indicated in Fig.10. The impeller may have to be tilted to insert it through the stirrer port.

Viewing from above, adjust the stirrer on its mounting rod so that the impeller is axially centered at its vertical position in the mixing chamber.

- if not already fitted, press a stainless steel insert into one end of the Teflon-lined PE sample input line and make input and output connections to the sample pump as indicated in Fig.11 using the appropriate size wrenches (5/8" and 11/16"). Make certain to brace movement of the "T" fittings with the wrench while tightening the nuts of the sample lines so that the position of the "T" fittings is not altered (the pulse suppressors must be near-vertical for proper operation). Insert a glass sampling wand into the intake end of the sample input line using a twisting motion.

- the pumps should have been set at the estimated settings for a 50L sample processed at 500 mL/min. If not, adjust them according to the settings in Table I. Some "fine adjustment" of these settings may be necessary during the first few extractions (see "RUN-IN").

When not in use, the extractor should be filled with high purity water and the outlets protected from dust with aluminum foil pieces.

"RUN-IN"

Before proceeding with the extraction of "real" samples, the extractor should be cleaned by extraction of several samples of high purity water (known to be free of the contaminants being determined, considering the sample size). This will serve as well to give the operator some practice in operation of the extractor and to make fine adjustments in pumping rates for the sample size and sample input rate desired.

The example given refers to the extraction of a 50 L sample at a sample input rate of 500 mL/min. Most early validation work was done using similar conditions. The apparatus, however, is designed with the capability to operate up to 1000 mL/min and the user is encouraged to experiment to find the appropriate conditions for the particular application.

- adjust the sample pump input rate to 500 mL/min by calibration using a 1 L graduated cylinder and stopwatch.
- add 300 mL of the extractant, dichloromethane (DCM), to the extractor through the scrubber port and when the DCM level has settled (through a dry column, almost all of this DCM will pass into the extractor), mark its position on the outside of the mixing chamber using a waterproof ink marker.
- begin extraction (see **OPERATION**) of a representative sample size (50 L) of high purity water.
- during the first 30 min of operation, check the solvent "make-up" and surrogate standards delivery lines (eg. insert an air bubble) to ensure that they are delivering liquid.
- after 30 min, stop the extraction and note the level of DCM. If below the mark, add DCM until the level is at the mark, then increase the solvent "make-up" pumping rate. If above the mark, drain DCM until the level is at the mark, then decrease the solvent "make-up" pumping rate.

When adding DCM to the system through the "scrubber" port while it is full of water or while the system is operating, pour the solvent slowly to prevent it from flowing or being carried over into the separator trap.

- continue the extraction.

- at 60 min, stop the extraction again and check the DCM level. Repeat the above adjustments if necessary.

- at 90 min, stop the extraction and repeat the above adjustment process if necessary. At this point, 10 min are left in the extraction of 50 L if the sample pump is correctly set. Additionally, the 100 mL of surrogate standard solution should have been delivered by this time (last 10% of the extraction time), allowing time for rinsing the standards container, pump-head and transfer line. If these conditions are not met, adjust the pumps accordingly and finish the extraction.

- practice the extract collection process (see **OPERATION**) then drain all components of the extractor (extractor body, separator trap and heater tube).

- repeat this "run-in" procedure with at least two more 50 L samples of high purity water and until all pump settings are seen to be correct for this sample size and sample input rate, (ie. the DCM level in the extractor should be maintained near the "300 mL" mark through the extraction, the 100 mL surrogate standards solution should be delivered with 10 min allowed to rinse the delivery system during the last 10% of the extraction, and the sample input rate should be well calibrated ($N \geq 5$) at 500 mL/min).

OPERATION

Given here is an operating procedure for processing a 50 litre sample of relatively "clean" aqueous sample (ie. low concentrations of dissolved organic matter) which has been clarified of particulate matter. Operation in this application and with this sample size has been thoroughly validated (4,5,6,9).

For operation with more complex matrices, different suites of contaminants, sample sizes and processing rates, the user is referred to more comprehensive applications literature (10,11) and to the list of documentation provided in this manual (Appendix B). If this applications material is insufficient for the intended application, the user, necessarily, must conduct the required research, development and validation.

Extractions are generally conducted at 20°C as most physical constants (eg. octanol-water partition coefficient, K_{ow}) describing solvent extraction processes are derived from data at or near this temperature. Use of the heater tube accessory, then, depends on sample temperature and must be adjusted to bring the sample temperature to 20°C. If the sample temperature is above 20°C, the accessory is not used and the ambient sample temperature is reported.

Control of dispersion is perhaps the most critical aspect of extraction with the GLSE. The advantage with GLSE prototypes is that dispersion is, controllable. The solvent/water dispersion must be restricted primarily to the mixing chamber; this is accomplished by proper adjustment of the stirring speed at a particular sample input rate. A significant flow of solvent should be allowed to recirculate to the mixing chamber through the first solvent recirculation arm, with a minimal amount reaching the second recirculation area, the settling chamber. The "instinct" to maximize dispersion (by stirring at an excessive rate) in order to increase mass transfer of analyte from sample to solvent must be avoided as this serves only to increase analyte loss by the escape of fine droplets of solvent (containing analyte) through the "scrubber" column and separator trap. With some matrices, high in content of dissolved organics or gases, this loss may be so significant as to exceed the solvent "make-up" provision and, if attention is not paid to the extraction, an excessive amount of extract may be lost, making a re-sample necessary.

General

Using a sink tap, provide sufficient dilution water (5-10 L/min) in the drain for dilution of the extraction effluent (see **PRE-INSTALLATION** for comments on disposal of extraction effluent).

Ensure that all drain lines actually feed to the drain and that they are unobstructed (ie. no "kinks", not compressed by other equipment).

Ensure that all stopcocks on the GLSE are closed.

Ensure that stirrer, heater (if to be used) and pump power switches are in the "OFF" position, and that power is switched "ON" at the power bar main switch (Fig.1).

Ensure that there is sufficient DCM in the solvent "makeup" container for the extraction and that the "pick-up" tube is in place.

If on-line surrogate standard addition is being used, ensure that the surrogate standard solution is at room temperature, then fill the standard addition container (100 mL graduated cylinder) with solution and insert the "pick-up" tube in the solution.

Sample volume, which must be measured and reported to the analytical laboratory for calculation of concentrations, may be estimated by reliance on sample container calibration. Alternatively, the sample volume may be estimated by timing the extraction period (from the moment sample enters the extractor to the moment water stops entering the extractor) and calculation, using this measurement and the calibrated sample

input rate. If the calibrated container method is used, a correction must be made for the volume of the heater tube and sample input line (~300-400 mL).

Procedure

1. Dry the floor of the extractor case before conducting an extraction. This makes visual determination of leaks in connections, tubing, glassware and at stopcocks much easier.
2. Drain the extractor components of storage water using the Tygon tubing drain lines.
3. Close all stopcocks.
4. Add ~300 mL of DCM to the extractor through the "scrubber" port directly from the solvent container or from a cleaned container reserved for this purpose only.
5. Fit a cleaned sampling "wand" in the end of the sample input line, then insert the "wand" in the sample.
6. Turn on the sample pump.

7. If estimating sample volume by timing the extraction period at a calibrated sample input rate, start timing as soon as sample begins flowing into the mixing chamber from the heater tube. At the same moment, turn on the solvent "make-up" pump, the surrogate standards pump and the heater (if to be used) (see General, above).
8. After a few hundred mL of sample has entered the extractor (the extractor will be $\sim 1/3$ to $1/2$ full), start the stirrer.
9. After 5-10 min of operation, make any adjustments to stirring speed necessary to confine the majority of solvent/water dispersion to the mixing chamber with some solvent return in the first solvent recirculation arm and minimal dispersion to the second recirculation chamber.
10. After 20 min of operation, stop the extraction (pause the timing device) and check the DCM level. Make adjustments as necessary (see "RUN-IN").

With some matrices (eg, high dissolved organics, dissolved gases) solvent consumption may be increased above that due to its solubility in water, making an increase in solvent "make-up" necessary.

11. Re-start the extraction (continue the timing device). At this point, assuming that an effective "run-in" has been done, there should be no need to attend the extraction until 90 min, at which point the surrogate standards container should be rinsed with ~5 mL of methanol to "flush" the standards distribution system.
12. As the sample is completed, raise the sampling wand above the level of the extractor inlet and as air begins to enter the heater tube, stop the timing and record the extraction time (min & s, convert to min).
13. Turn off all components except the stirrer. Continue stirring for at least 2 min to extract the last portion of sample entering the extractor.
14. Turn off the stirrer and allow the phases to separate and clear.
15. Before collecting extract, give the stirrer a few momentary "bursts", or stir slowly for several minutes, if necessary, to assist in clearing the DCM phase.

Extract collection

16. When phases have cleared sufficiently, collect, using the **extract drain**, the bulk of the extract directly into the extract container, including any "scum"

which has formed at the DCM/water interface. Collect any DCM caught in the sample drain (Fig.4) or which has been carried over into the separator trap (separator trap drain, Fig.5) directly into the extract container.

Ideally, with proper control of the degree of phase mixing, no DCM should be carried over into the separator trap. With some matrices (high concentrations of organics, dissolved gases, dissolved inorganic, or high loadings of fine silt, colloidal material, etc.), this may be unavoidable. Other techniques of dealing with poor separation and clearing of phases are dealt with in (10).

17. Drain the separator trap using a Tygon tubing drain line connected to the separator trap drain.
18. Drain ~1/2 of the water remaining in the extractor to the sink drain using the sample drain and Tygon tubing.

As water drains from the "scrubber column", DCM which has collected in the column will fall out until the water level is below the column. In draining the extractor, be careful not to drain so fast that this DCM is carried out through the drain line.

19. Using the 500 mL separatory funnel supplied with the GLSE, collect DCM which has fallen out of the column and a sufficient amount of extracted water to fill the funnel.
20. Add this DCM to the extract container and pour $\sim 1/2$ the water in the separatory funnel back into the extractor through the "scrubber" column to flush out remaining DCM.
21. Continue this draining and column flushing process using the separatory funnel. With each collection, add any DCM collected to the extract container and use a portion of the water to flush the "scrubber column". The column should have been flushed 3-5 times during this process.
22. Before the water level in the mixing chamber has fallen below the impeller, momentarily activate the stirrer to dislodge any DCM which has collected on the upper surface of the impeller.
23. Ensure that the DCM extract in its container is layered with $\sim 1/4$ " of extracted water. This serves to minimize evaporation of DCM during storage.

24. A useful measure at this point is to mark the phase meniscus (water/DCM, water/"scum"/DCM) on the outer surface of the extract container with a "permanent" ink marker (cover mark with transparent tape) or with similar marking on a strip of "lab tape" placed vertically on the container. This marking may later be useful in assessing extract loss during transport and storage and in noting the "clearing" of "scum".
25. Cap and label the extract container and store refrigerated. Re-tighten the cap a few hours later as it may loosen on cooling.
26. Close all stopcocks on the GLSE apparatus and fill the apparatus with "high purity" water.

Intersample Cleaning and Short-Term Storage

If the extract is collected as described in this section, analyte loss due to failure to collect all extract in the GLSE should be minimized to 0-5 mL of extract (ie. < 2% of the total extract collected). It is impractical to attempt to collect this remaining few % of "lost" analyte; the effect will be accounted for in the surrogate standards correction. The intersample cleaning procedure should minimize any significant effect on the subsequent sample.

For relatively "pristine" matrices, intersample cleaning of the extractor is limited to filling it with "high purity" water, stirring for 10 min, then draining the water. Alternatively, the extractor body may be rinsed by stirring an "extractor-full" of high purity water with 300 mL of DCM or the entire apparatus may be rinsed by processing 10L or more of high purity water as though it were a sample. More rigorous cleaning methods are discussed in (10).

PRECAUTIONARY ADVICE

Although application of the GLSE with some aqueous matrices and certain classes of contaminants has been well- studied, the technique remains a research tool and successful application depends on the extent to which the user has done the necessary background investigation (ie. "homework") and is willing to perform the required development and validation work in cases where documentation does not exist.

SAFETY/HAZARD INFORMATION

The extractant (DCM) is a hazardous substance and all personnel working in the area of the GLSE during operation must be so informed by the provision of the appropriate WHMIS and MSDS information (APPENDIX C).

Government regulations (municipal, provincial, federal) must be consulted for limits applicable to the disposal of extraction effluent (see **PRE-INSTALLATION**).

The power distribution bar should be located so that it will not be wetted by activities associated with operation of the GLSE.

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TABLE I : OPERATIONAL SETTINGS

Component Description	Setting	Comments
SAMPLE PUMP (FMI Model # QD 2CSC)	4.2	500 mL/min (nominal)
SOLVENT "MAKE-UP" PUMP (FMI Model # RRP SY 2CSC)	1.4	6.50 mL/min (nominal)
SURROGATE STANDARDS PUMP (FMI Model # RP SY 0CSC)	2.2	1.11 mL/min (nominal)
STIRRER Caframo Model # RZR50	800-1000	Setting approximates RPM and is dependent on phase mixing characteristics (See OPERATION)
HEATER CONTROLLER Cole-Parmer # (Used with Corning Model No. Silica Immersion Heater)	0-100%	Setting depends on sample temperature (eg. 40% for sample at 5°C processed at 500 mL/min to reach 20°C)

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APPENDIX I

PARTS LIST FOR THE GOULDEN LARGE-SAMPLE EXTRACTOR
(LASALLE SCIENTIFIC INC. PACKAGE)

ITEM	DESCRIPTION	MINIMUM QUANTITY	SUPPLIER	LASALLE PART NUMBER
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GLSE-95				
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MAJOR GLASSWARE COMPONENTS

Extractor Body	1	LASALLE SCIENTIFIC INC.
Separator Trap	1	121 Malcolm Rd.
Accessory Tube	1	Guelph, Ontario N1K 1A8
		(519) 824-7301
		FAX (519) 824-9576

MINOR GLASSWARE COMPONENTS

Spiking Inlet (Set/2)	1
Spike Pick-up Tube (Set/4)	1
Solvent Delivery Tube (Set/4)	1
Solvent Pick-up Tube (Set/4)	1
Vent Tube (Set/4)	1

A2

Pulse Suppressor (Set/2)	2
Sampling Wand (Straight) (Set/6)	1
Sampling Wand (Right-angle) (Set/6)	1

EXTRACTOR CABINET

LASALLE SCIENTIFIC INC.
(above)

ELECTRICAL/MECHANICAL COMPONENTS

Power Outlet	6 outlets, independently switched, independently fused, 115VAC/15A	1
Immersion Heater	Silica, 115VAC/15A, 1000W	1
Power Controller	SCR, 115VAC/15A, 1000W	1
Stirrer	High torque, constant torque, chuck size to 10 mm.OD, suitable for continuous operation, variable speed to 2000 rpm	1
Pump (Sample)	Positive displacement, high precision delivery (± 1 % or better), non-contaminating and chemically inert wetted components, suitable for continuous operation, 0-1000 mL/min.	1

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Pump (Solvent Compensation)	(") 0-45 mL/min.	1
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Pump (Standard Addition)	(") 0-5mL/min.	1
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PUMP FITTINGS

Male Run Tee	316 Stn.Stl., 3/8" tube OD to 1/4" male pipe (NPT) thread	2
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Male Elbow	316 Stn.Stl., 1/8" tube OD to 1/4" male pipe (NPT) thread	2
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Inserts	316 Stn.Stl., 3/8" tube OD to 1/4" tube ID	2
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Ferrules	Teflon, 3/8" tube OD, set of front and back ferrules	10
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Adapter	Teflon, low-flow isolation adapter for Stn.Stl. pump-heads, Set/2	1
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Small-bore fitting kit	1/4-28 fittings kit for 1/16" OD Teflon tubing	1
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LASALLE SCIENTIFIC INC.
(above)

MATERIALS

Sheet, Teflon	Teflon TFE (virgin), 1/32" thick, 12" * 12 "	1
Tubing, Teflon	Teflon TFE (virgin), 1/8" OD * 1/16" ID	6ft.
Tubing, Teflon	Teflon TFE (virgin), 5/16" ID, fractional spaghetti tubing, standard wall	15ft.
Tubing, Teflon	Teflon TFE (virgin), 3/16" ID, fractional spaghetti tubing, standard wall	1ft.
Tubing	Polyethylene, Teflon FEP lined, 3/8" OD * 1/4" ID, Penntube Plastics type "Pureline"	10ft.
Tubing, Silicone	4 mm. OD * 0.8 mm. ID (Size 13)	1ft.
Tubing, Silicone	1/4" OD * 1/8" ID	1ft.
Tubing, Tygon	9/16" ID * 3/32" wall, Norton Performance Plastics type R-3603	10ft.

A5

Tubing, Tygon	3/8" ID * 3/32" wall, Norton Performance Plastics type R-3603	10ft.
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MISCELLANEOUS PARTS

Clamp	Micro, Polymer Tech Model No.58858	1
Clamp Holder	Lab-Line Model No.7040	3
Clamp Holder	Jumbo, Polymer Tech Model No. 58875	1
Base Foot	Lab frame	4
Rod	Aluminum, hardened, corrosion- resistant, 1/2" diameter, 36" length cut to 26"	1
Stirring Paddle	3 bladed, 2" diameter, 10" length (eg. Talboy's Engineering Corp. Model No. 151)	1
Separatory Funnel	Teflon FEP, 500 mL, "Nalgene"	1

A6

Support Ring	Extension, "Humboldt" Model No. H18440	2
Thermometer	Dial type, -10 to +100°C	1
Hose clamps	5" OD (nom.)	3
	3 & 1/2" OD (nom.)	1
	2 & 1/2" OD (nom.)	3
	1/2" OD (nom.)	2

A7

GLSE-95-PKG

Complete GLSE system, including
all the above components, assembled
and mounted in a transportable
cabinet

1 LASALLE SCIENTIFIC INC.
(above)

EXTRACT PROCESSING GLASSWARE

Goulden Evaporator I Distillation of dichloromethane
Fig. 12
(References 8,10)

1

LASALLE SCIENTIFIC INC.
(above)

Goulden Evaporator II Distillation of hexane
Fig. 13
(References 8,10)

1

B1

"APPENDIX B"

**DOCUMENTATION OF GOULDEN LARGE-SAMPLE EXTRACTOR (GLSE)
TECHNOLOGY:**

DEVELOPMENT, VALIDATION AND APPLICATIONS STUDIES

From:

Anthony, D.H.J. 1993. "Incorporation of Goulden Large-Sample Extraction Technology in Water Quality Monitoring and Research Programs", National Water Research Institute, Contribution No. 92-.

DOCUMENTATION OF GOULDEN LARGE-SAMPLE EXTRACTOR (GLSE) TECHNOLOGY:

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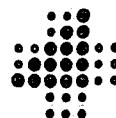
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Material Safety Data Sheet

emergency telephone no. 312/973-3600 (American Scientific Products)
 chemtrec telephone no. 800/424-9300
 information telephone no. 616/726-3171 (American Burdick & Jackson)

MATERIAL SAFETY DATA SHEET

I. Identification

chemical name Methylene Chloride molecular weight 84.93
 chemical family Chlorinated Hydrocarbon formula CH₂Cl₂
 synonyms Dichloromethane, Methylene Dichloride
 DOT proper shipping name Methylene Chloride or Dichloromethane
 DOT hazard class ORM-A
 DOT identification no. UN1593 CAS no. 75-09-2

METHYLENE CHLORIDE

(1,2-DICHLOROMETHANE)

II. Physical and Chemical Data

boiling point, 760mm Hg. 39.75°C freezing point -95.14°C evaporation rate (ether=1) ca 0.7
 vapor pressure at 20°C 350 mm Hg vapor density (air = 1) 2.9 solubility in water @ 20°C 1.6%
 % volatiles by volume ca 100 specific gravity (H₂O = 1) @ 20°C 1.33 stability Stable
 hazardous polymerization Not expected to occur.
 appearance and odor Clear, colorless liquid with a sweet ether-like odor.
 conditions to avoid Heat, sparks, open flame, open containers, poor ventilation, and moisture.

materials to avoid Active metals and strong alkaline solutions.

hazardous decomposition products Phosgene, hydrogen chloride, and chlorine.

III. Fire and Explosion Hazard Data

flash point, (test method) None (closed cup) auto ignition temperature 556°C
 flammable limits in air % by volume: lower limit 12.0 upper limit 19.0
 unusual fire and explosion hazards Concentrated vapors can be ignited by high intensity heat source or flame. Toxic and corrosive gases are formed on contact with flame or hot glowing surfaces.

extinguishing media Non-flammable material. Use dry chemical, carbon dioxide, foam, water spray as appropriate for surrounding fire and materials.
 special fire fighting procedures Non-flammable material. Wear full protective clothing and self-contained breathing apparatus. Heat will build pressure and may rupture closed storage containers. Keep fire-exposed containers cool with water spray.

IV. Hazardous Components

Methylene Chloride % ca 100 TLV 100 ppm CAS no. 75-09-2

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American Burdick & Jackson

Subsidiary of American
Hospital Supply Corporation

1953 South Harvey Street
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V. Health Hazards

<u>Occupational Exposure Limits</u>				<u>Concentration Immediately Dangerous to Health</u>	
OSHA	8-hour PEL	-	500 ppm	OSHA/NIOSH	5000 ppm
	Ceiling	-	1000 ppm		
	Peak	-	2000 ppm		
ACGIH	TLV-TWA	-	100 ppm	<u>Odor Threshold</u>	
	TLV-STEL (15-min)	-	500 ppm	OHS	200 ppm
NIOSH	TLV-TWA	-	75 ppm	NSC	200 ppm
	TLV-C (1-hour)	-	500 ppm		

Carcinogenic, Mutagenic, and Teratogenic Data

Indefinite animal carcinogen (IARC)
Experimental carcinogen (NTP)
Positive mutagen (RTEC)

Primary Routes of Entry

Methylene chloride may exert its effects through inhalation, skin absorption, and ingestion.

Industrial Exposure: Route of Exposure/Signs and Symptoms

Inhalation: Exposure can cause light-headedness, vertigo, drowsiness, narcosis, headache and dizziness, unconsciousness, and even death in extreme cases. Exposure to vapors can elevate carboxyhemoglobin levels in the cardiovascular system.

Eye Contact: Liquid or high vapor concentration can cause pain and irritation with slight corneal injury possible.

Skin Contact: Prolonged or repeated skin contact can cause irritation and dermatitis through defatting of skin.

Ingestion: Can cause burning of throat and mouth.

Effects of Overexposure

Acute inhalation or ingestion causes mild central nervous system depression. The primary toxic effect is narcosis. Other toxic effects are pulmonary edema, encephalopathy, and hemolysis. Methylene chloride irritates the eyes, skin and respiratory tract. No systemic effects have been reported in humans, although excessive concentrations have caused liver and kidney damage in animals.

Medical Condition Aggravated by Exposure

Preclude from exposure individuals with diseases of liver, kidneys; cardiovascular and central nervous systems, and heavy smokers. Simultaneous exposure to methylene chloride and alcohol can increase the toxic hazards of methylene chloride.

Emergency First Aid

- Inhalation:** Immediately remove to fresh air. If not breathing, administer mouth-to-mouth resuscitation. If there is no pulse, administer cardiopulmonary resuscitation (CPR). Contact physician immediately.
- Eye Contact:** Rinse with copious amounts of water for at least 15 minutes. Get emergency medical assistance.
- Skin Contact:** Flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothing and shoes. Wash clothing before re-use and discard shoes. Get emergency medical assistance.
- Ingestion:** Call local Poison Control Center for assistance. Contact a physician immediately. Never induce vomiting or give anything by mouth to a victim unconscious or having convulsions.

VI. Safety Measures and Equipment

- Ventilation:** Adequate ventilation is required to protect personnel from exposure to chemical vapors exceeding the PEL. The choice of ventilation equipment, either local or general, will depend on conditions of use, quantity of material, and other operating parameters.
- Respiratory:** Use approved respirator equipment. Follow NIOSH and equipment manufacturer's recommendations to determine appropriate equipment (air-purifying, air-supplied, or self-contained breathing apparatus).
- Eyes:** Safety glasses are considered minimum protection. Goggles or face shield may be necessary depending on quantity of material and conditions of use.
- Skin:** Protective gloves and clothing are recommended. The choice of material must be based on chemical resistance and other user requirements. Generally, neoprene or Buna-N offers acceptable chemical resistance. Individuals who are acutely and specifically sensitive to methylene chloride may require additional protective equipment.

Storage: Methylene chloride should be protected from moisture, temperature extremes, and direct sunlight. Proper storage of methylene chloride must be determined based on other materials stored and their hazards and potential chemical incompatibility. In general, methylene chloride should be stored in a cool, well ventilated and secure toxic storage room.

Other: Emergency eye wash fountains and safety showers should be available in the vicinity of any potential exposure.

VII. Spill and Disposal Data

Spill Control: Wear protective clothing and use approved respirator equipment. Absorb spilled material in an absorbent recommended for solvent spills and remove to a safe location for disposal by approved methods. If released to the environment, comply with all regulatory notification requirements.

Waste Disposal: Dispose of methylene chloride as an EPA hazardous waste. Hazardous waste number - U080(Toxic).

Revision Date: 1/85

KEY

ca	Approximately	STEL	Short Term Exposure Level
na	Not applicable	TLV	Threshold Limit Value
C	Ceiling	TWA	Time Weighted Average
PEL	Permissible Exposure Level	BuAc	Butyl Acetate

NSC National Safety Council ("Fundamentals of Industrial Hygiene", 1983)
OHS Occupational Health Services ("Hazardline")

Figure 1. GLSE-95 Components Layout

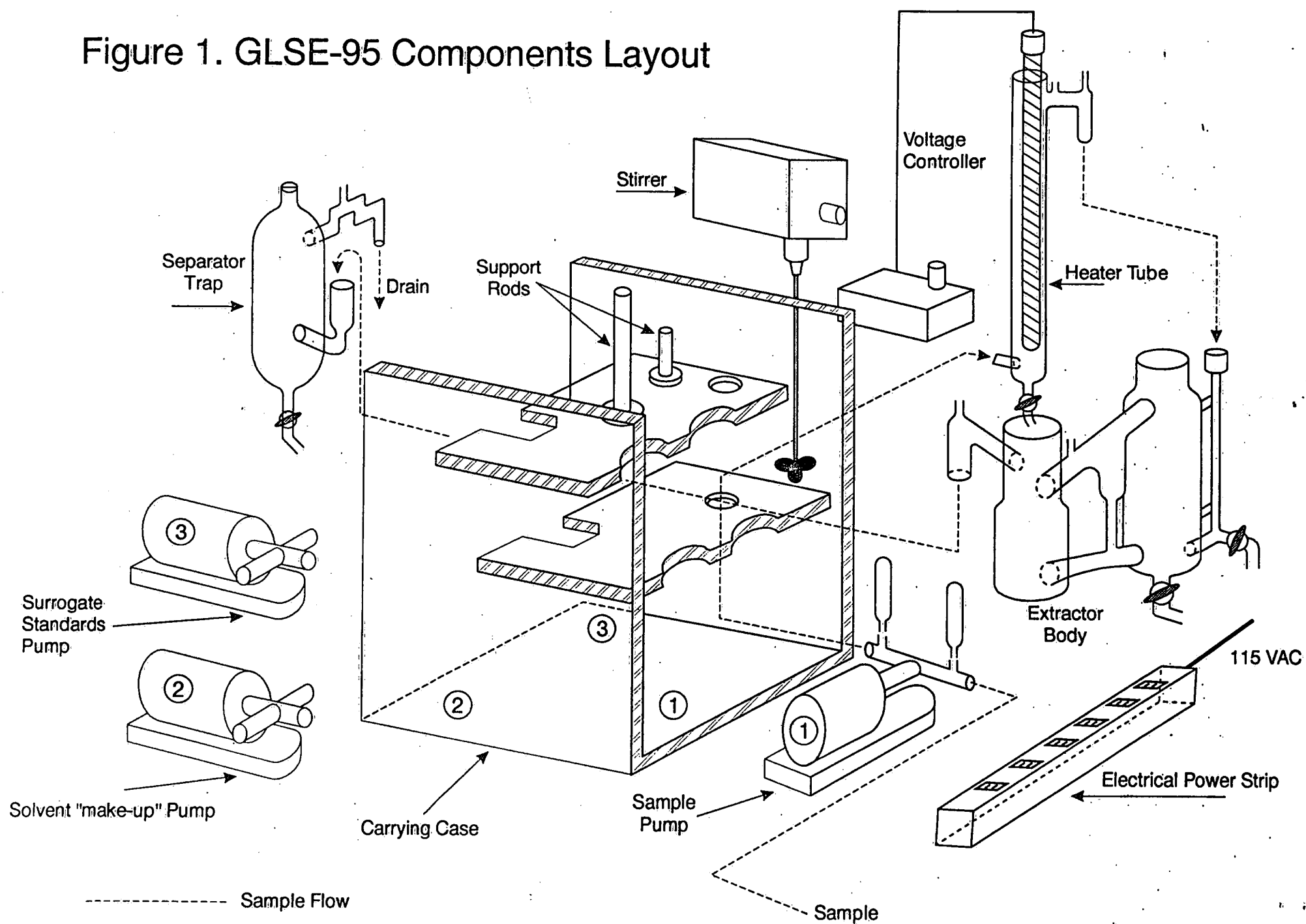


Figure 2. GLSE-95 : Major Glassware Components

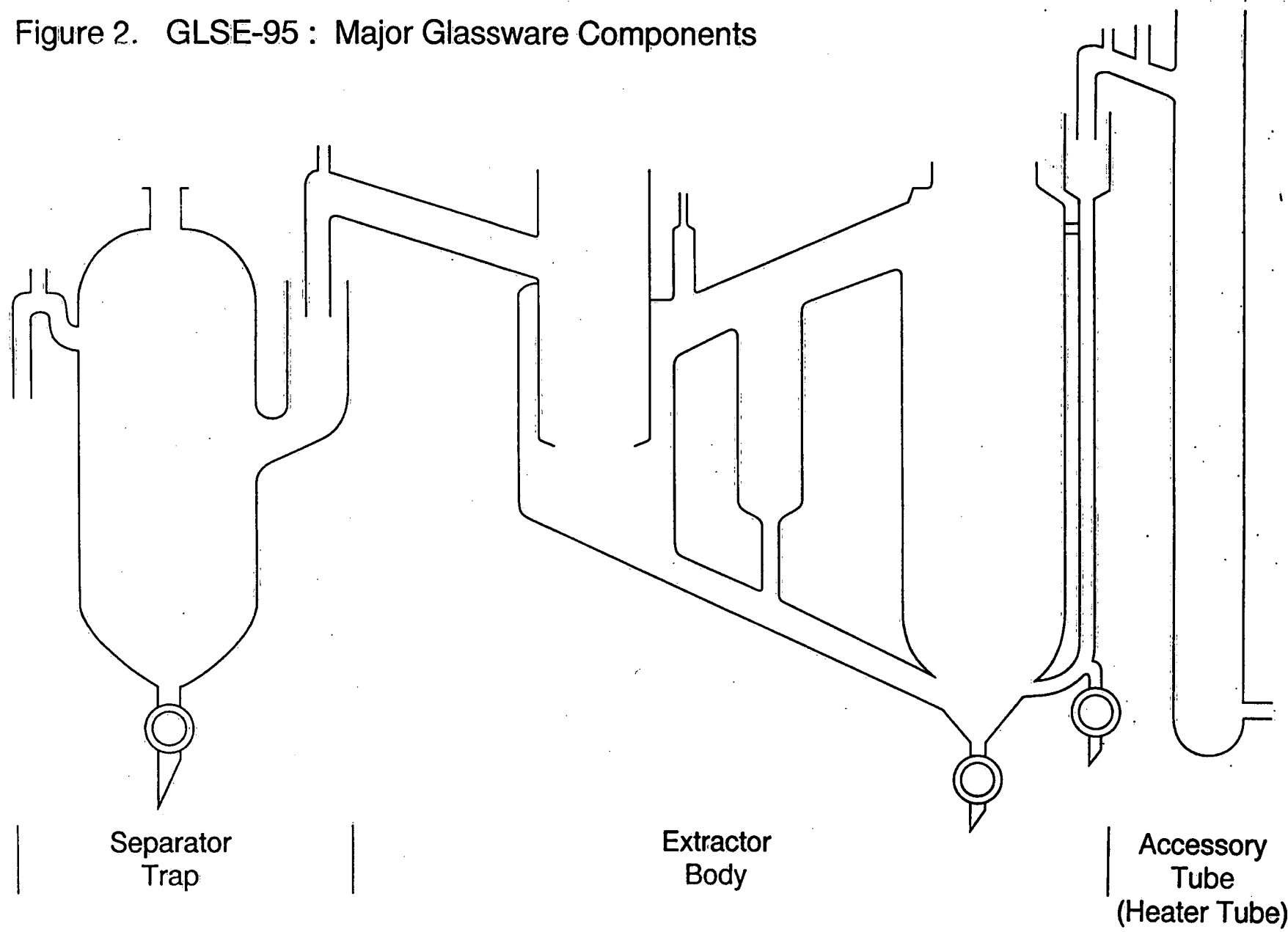


Figure 3. GLSE-95 Electromechanical Components and Fluid Flows

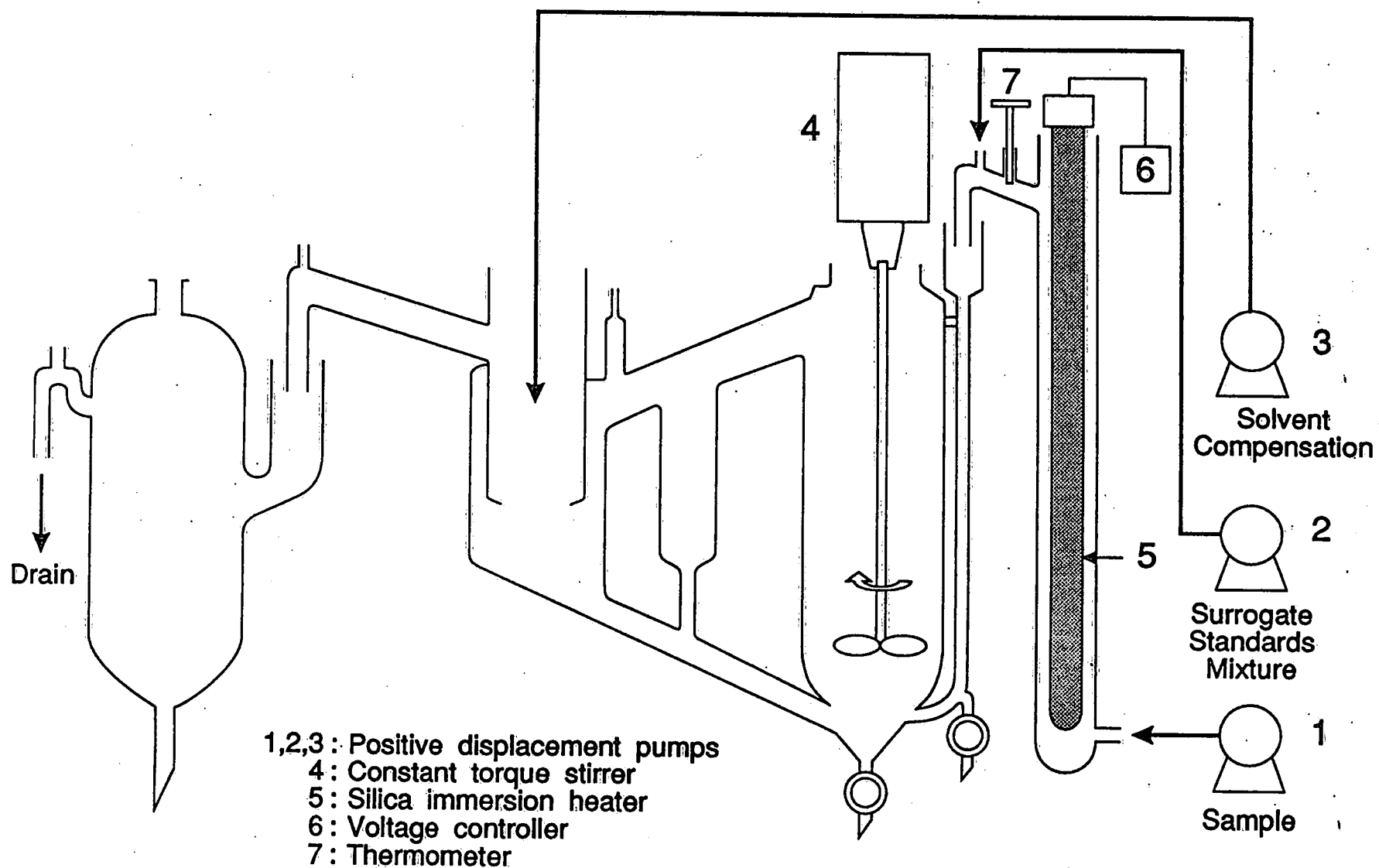


Figure 4. Extractor Body Details

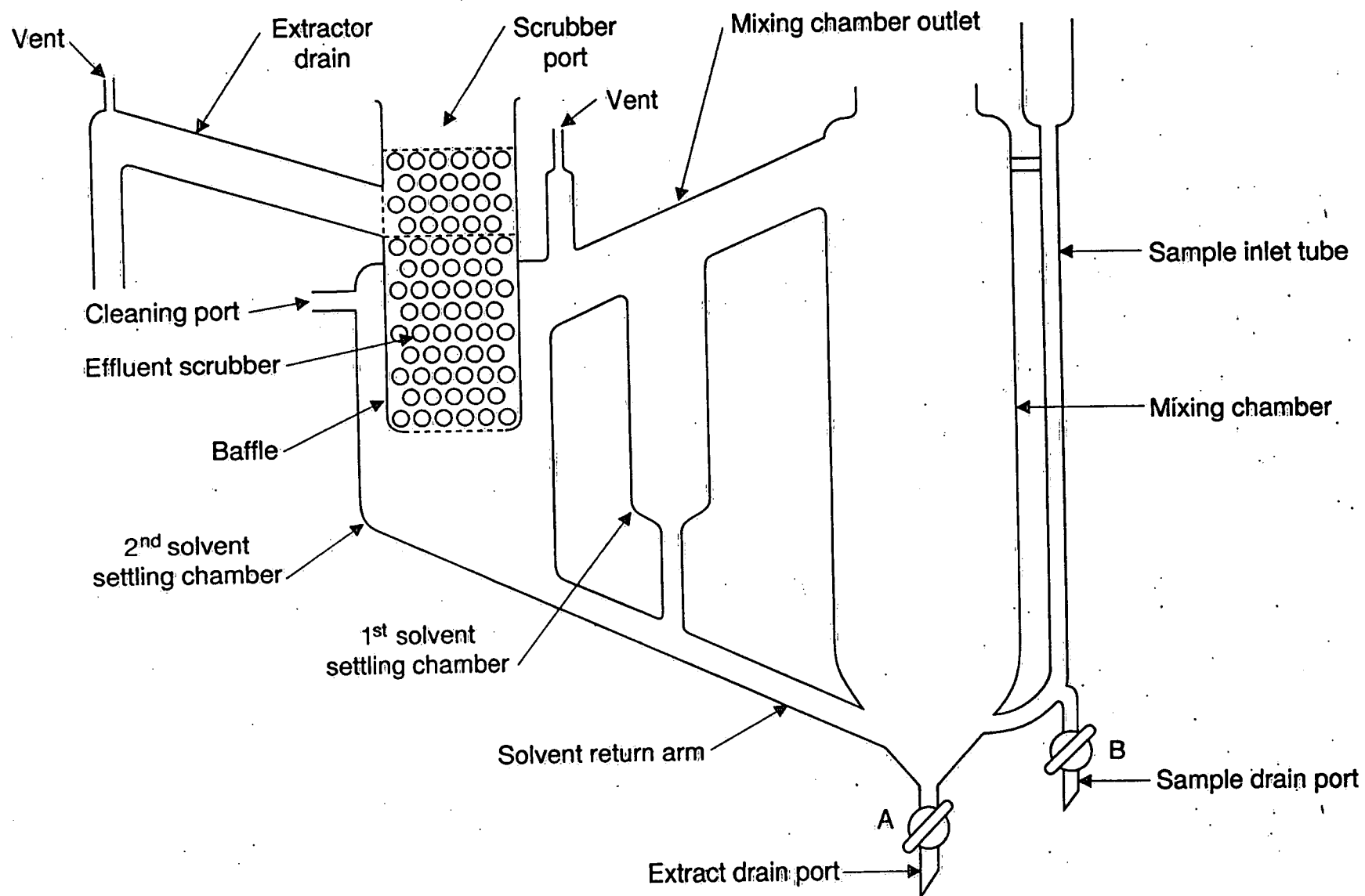


Figure 5. Separator Trap Details

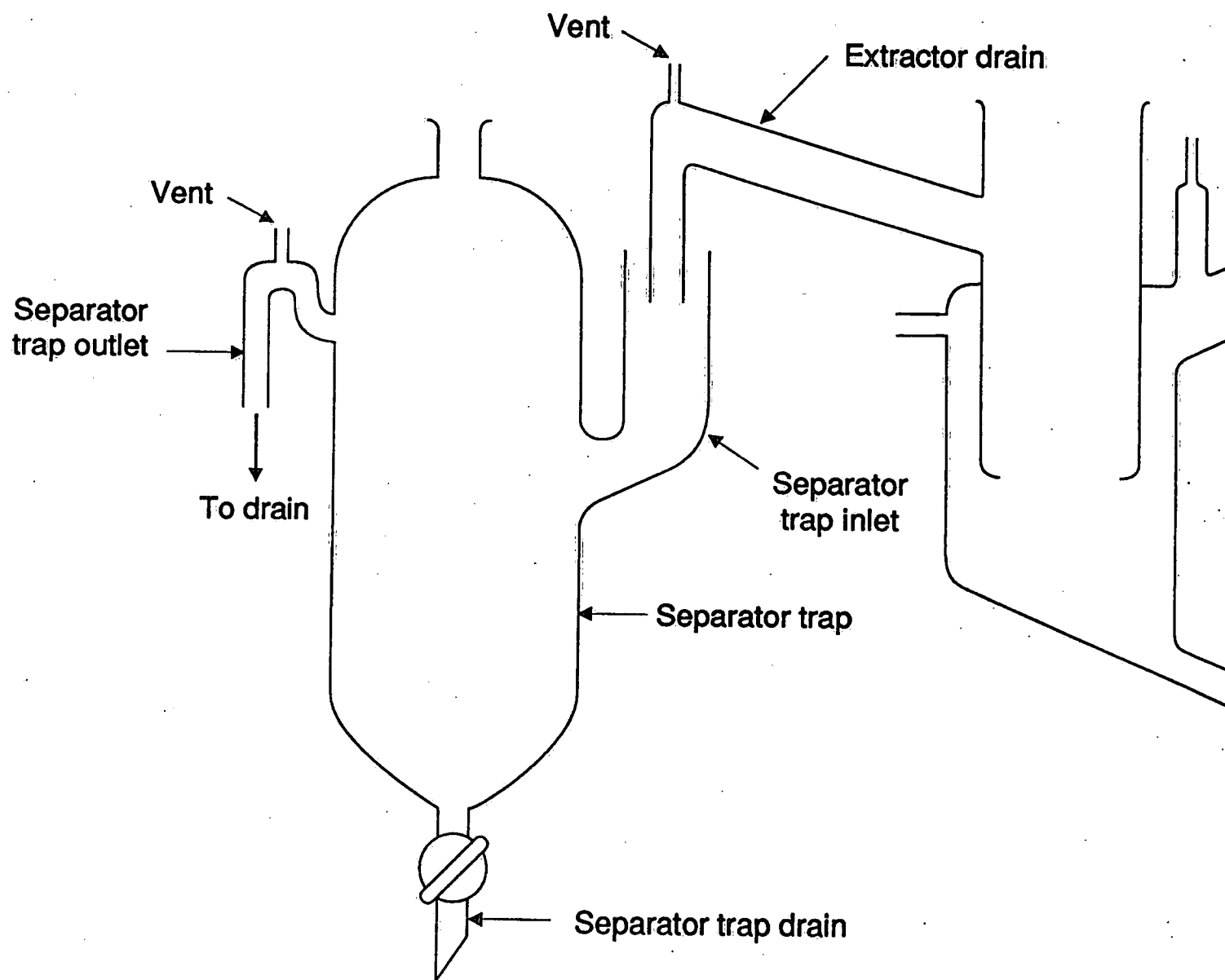


Figure 6. Heater Tube Details

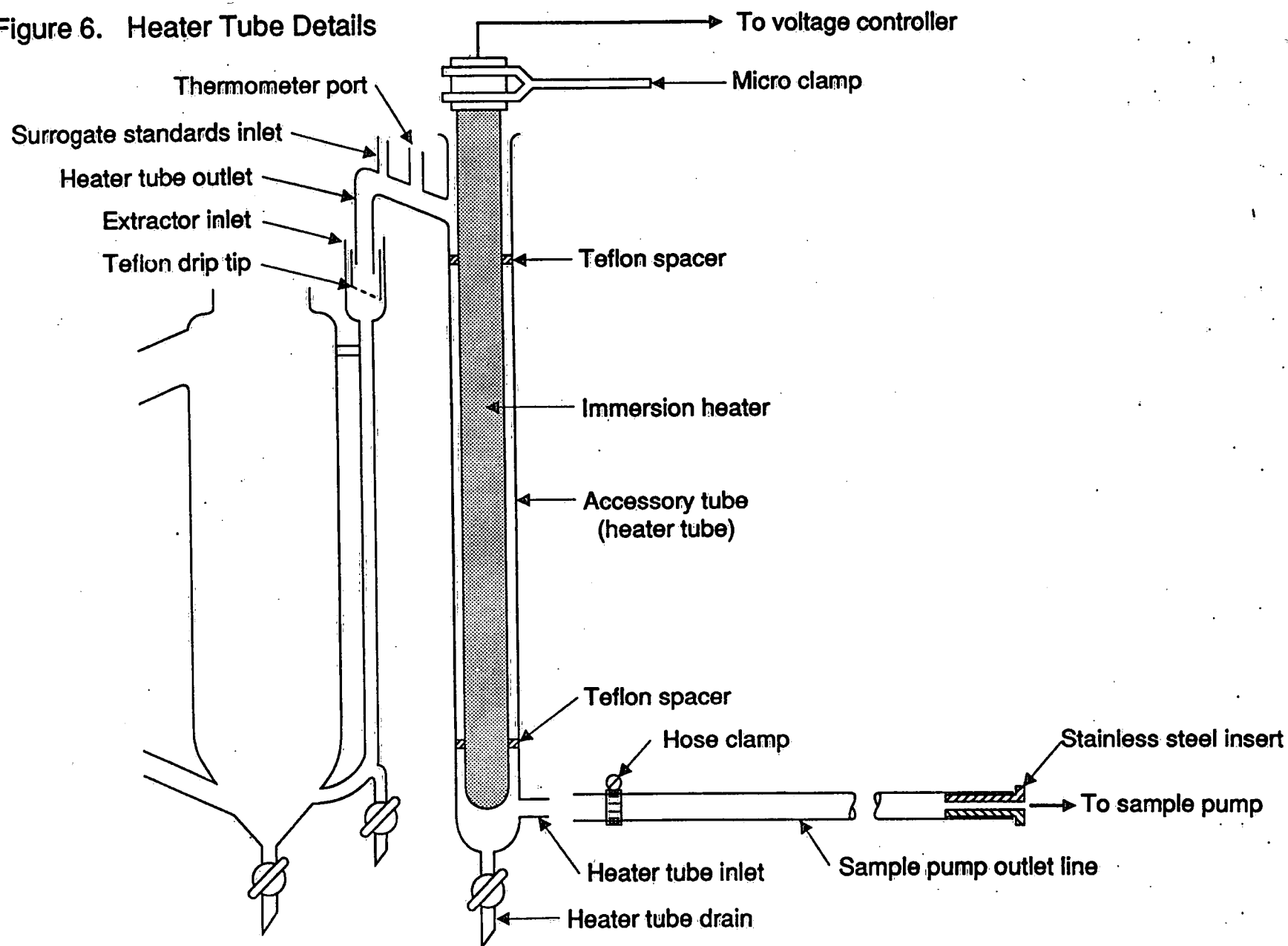


Figure 7. "Scrubber" Column Details

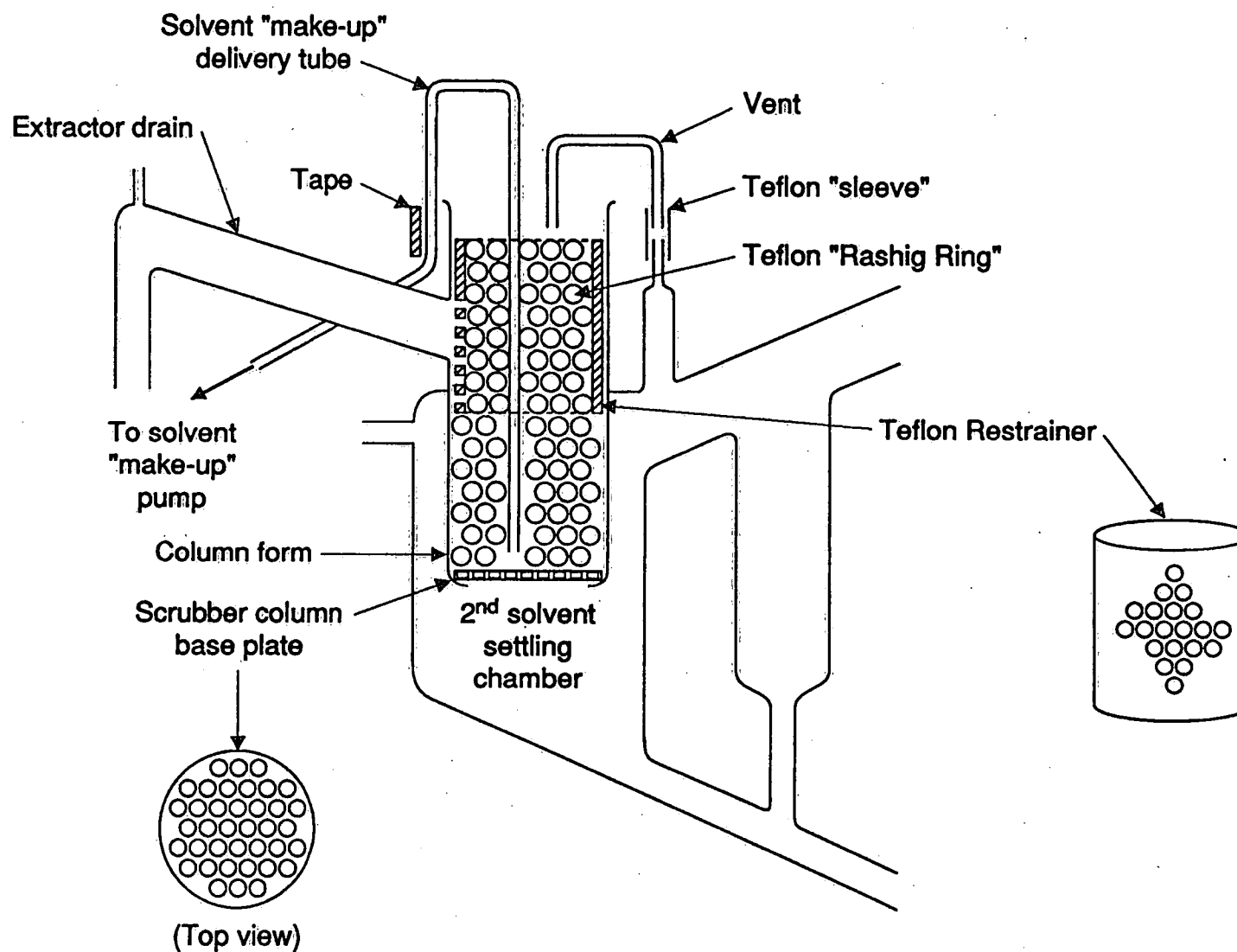


Figure 8. Solvent Compensation Delivery Details

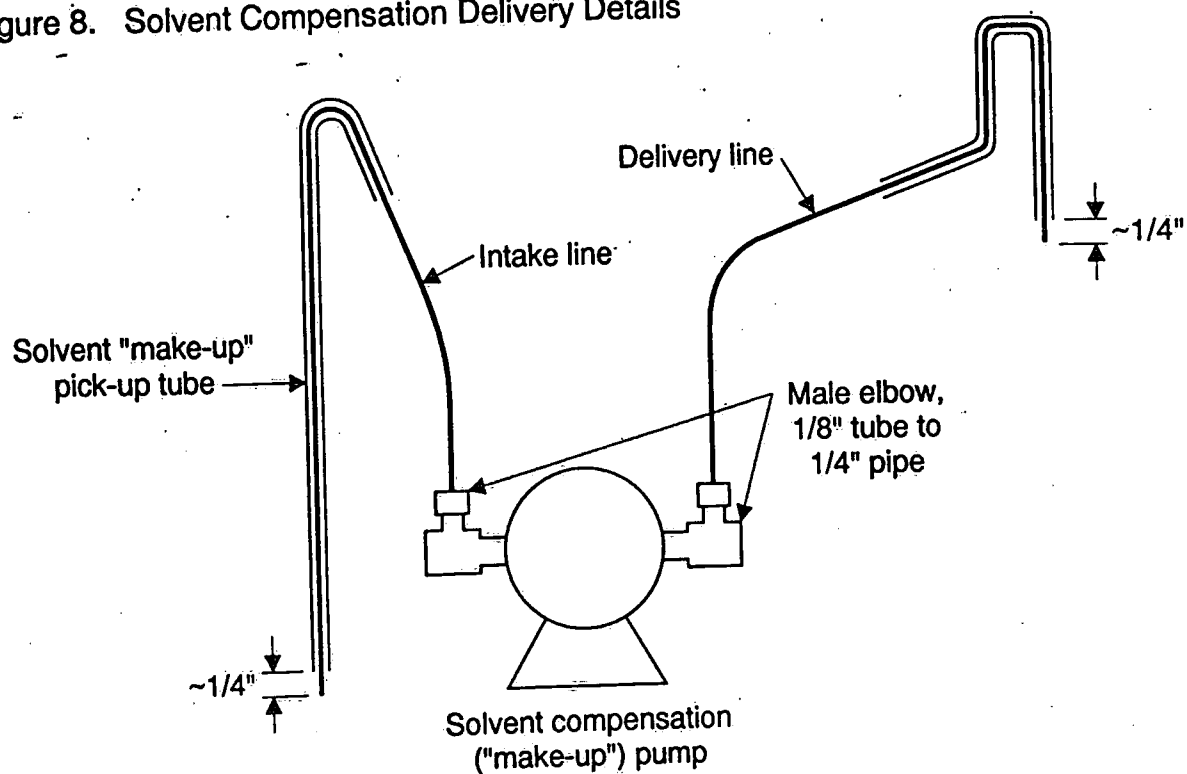


Figure 9. Surrogate Standards Delivery Details

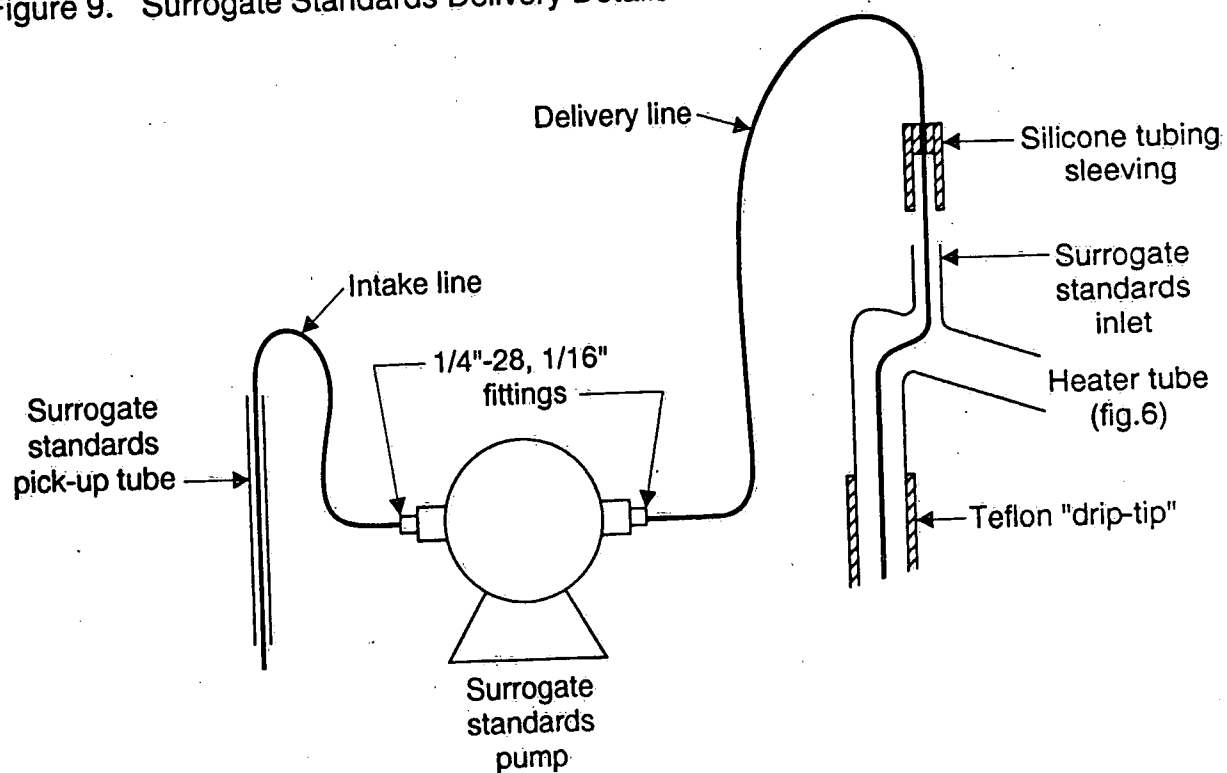


Figure 10. Impeller Positioning

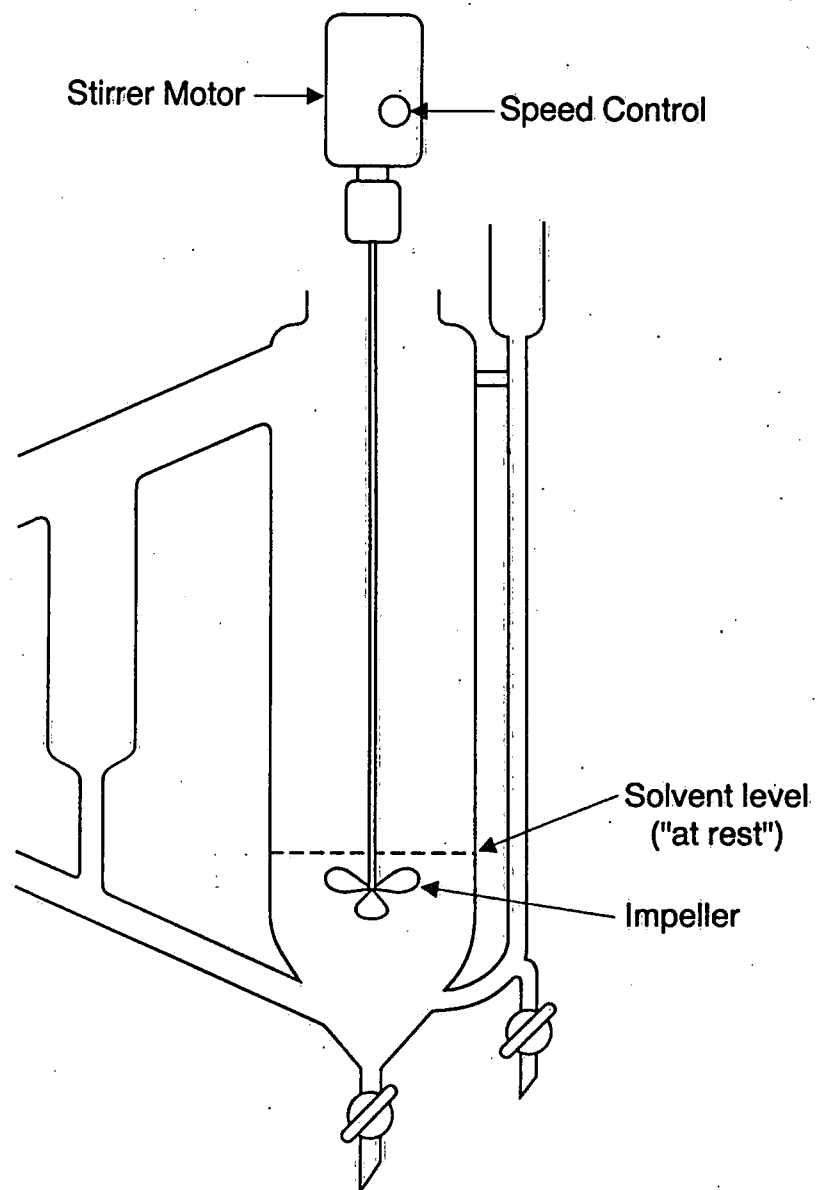
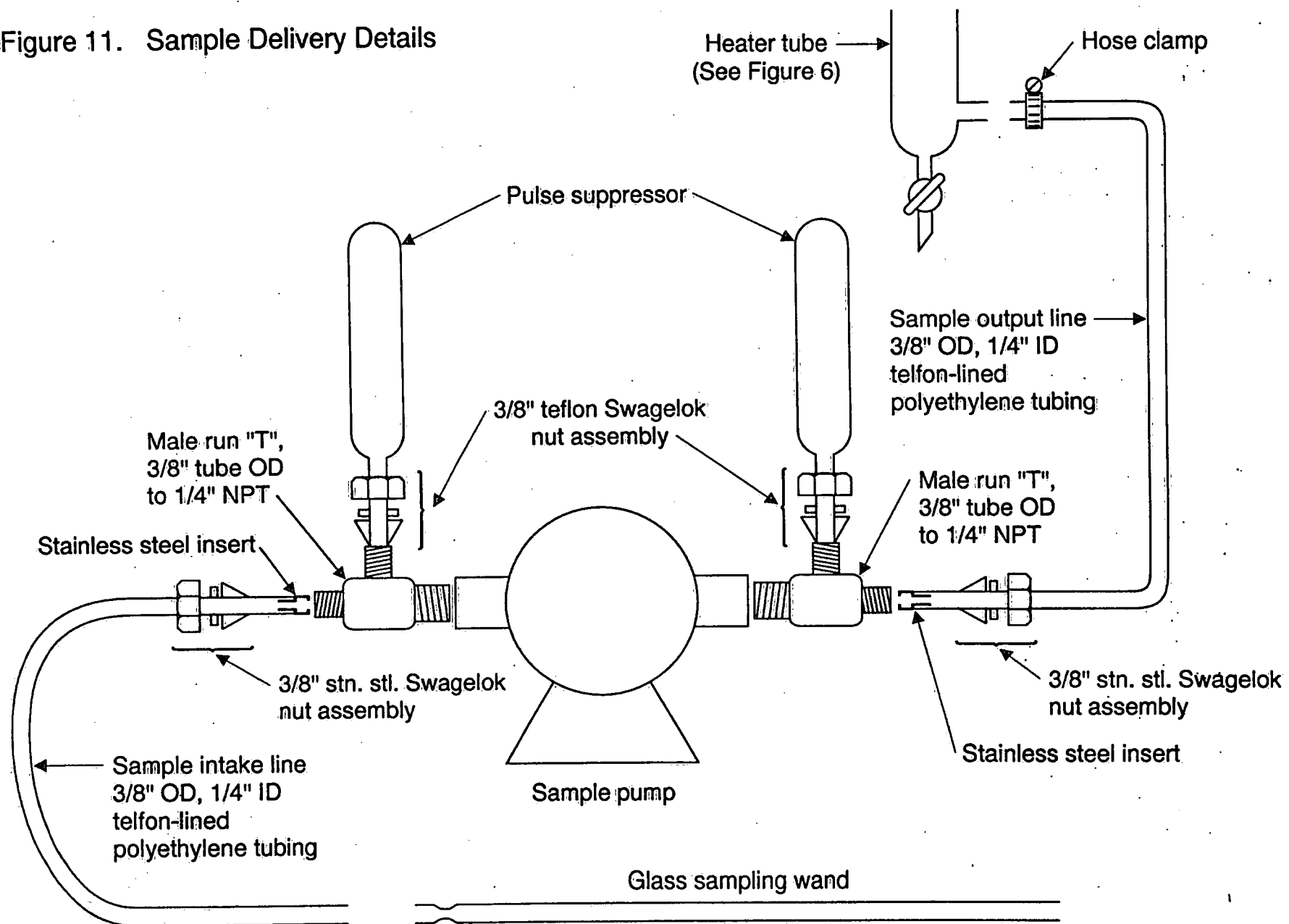
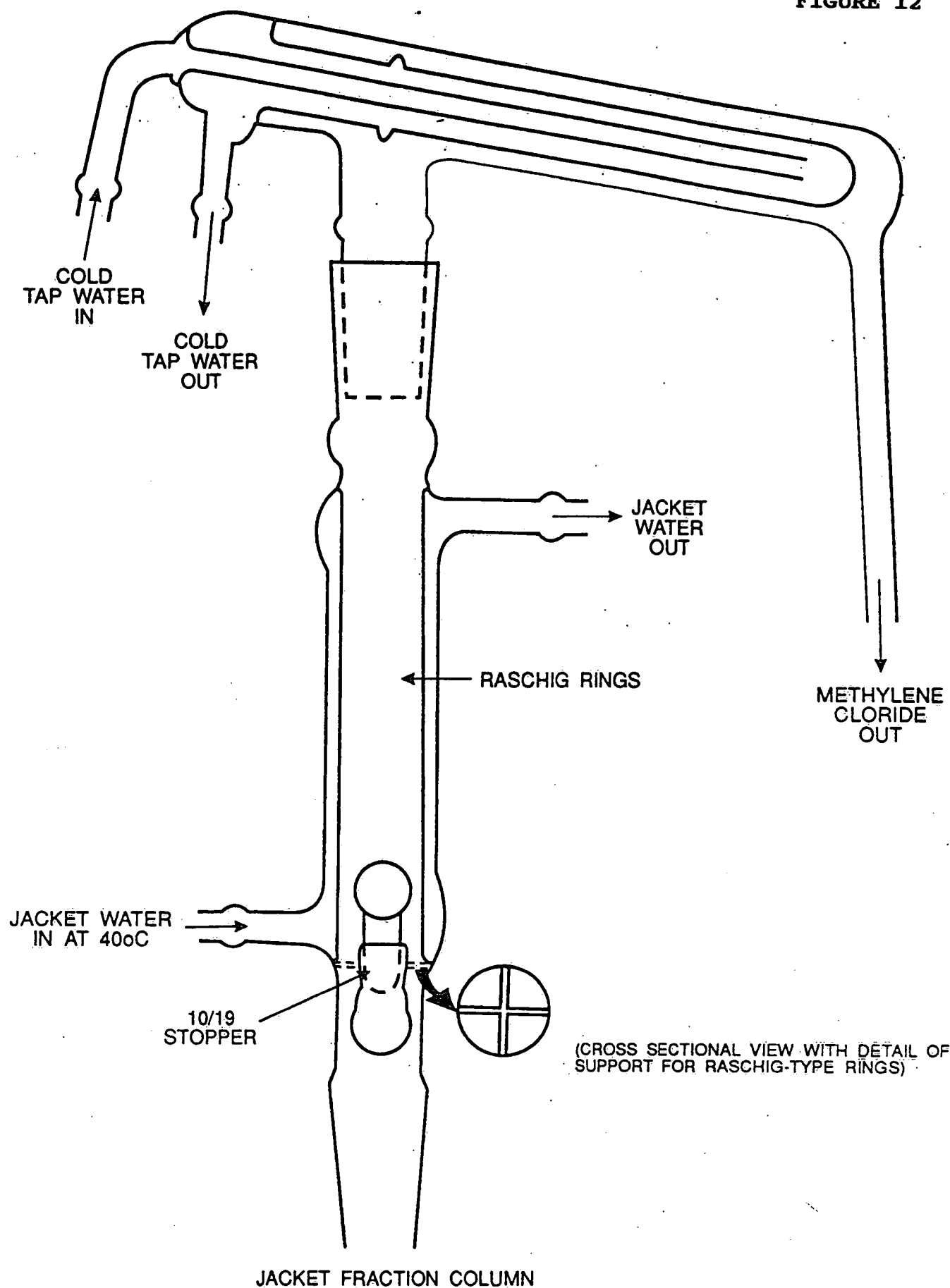


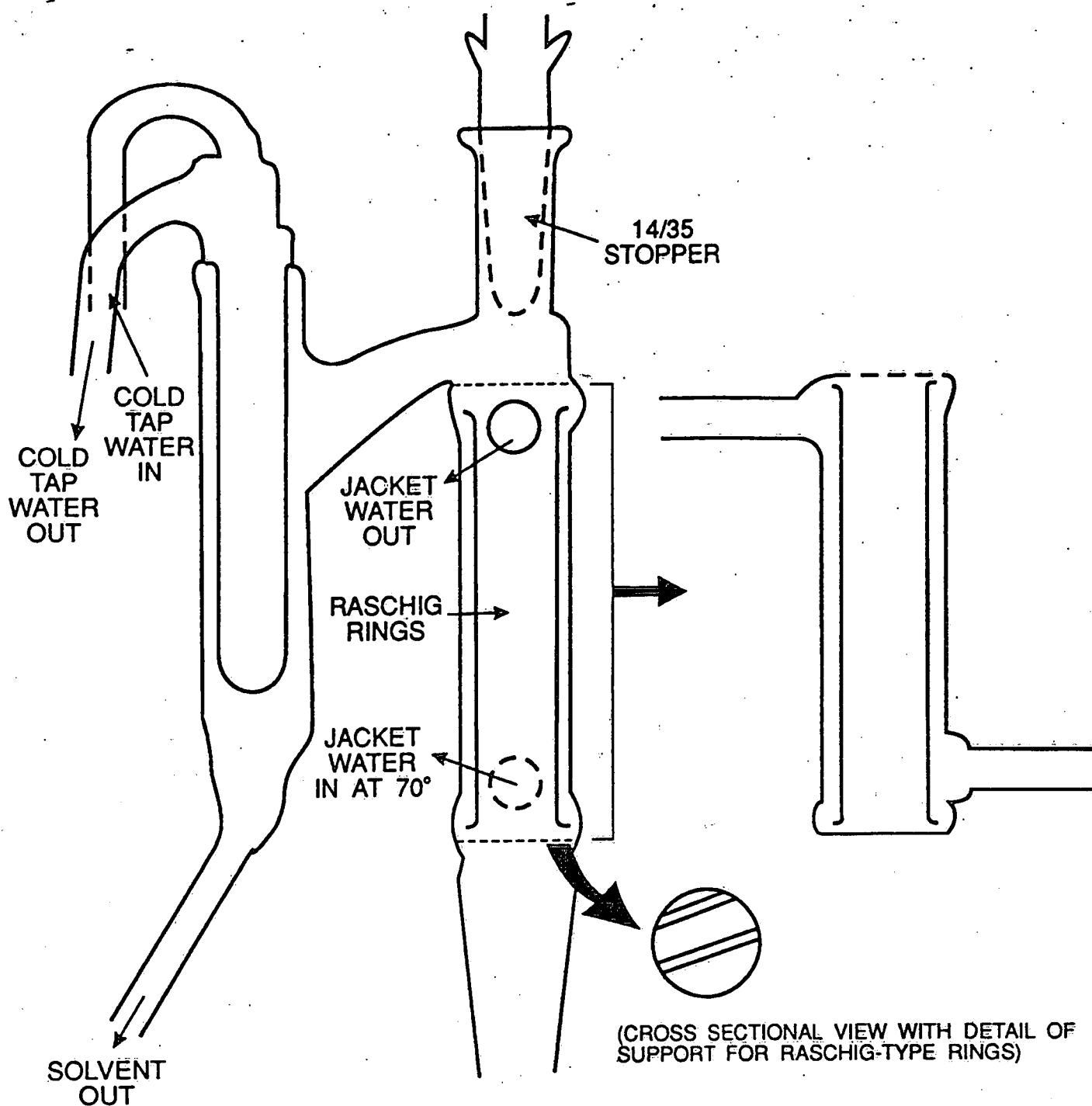
Figure 11. Sample Delivery Details





GOULDEN EVAPORATOR FOR DISTILLATION OF DICHLOROMETHAN

FIGURE 13



GOULDEN EVAPORATOR FOR DISTILLATION OF HEXANE

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3 9055 1016 6345 7

9945

