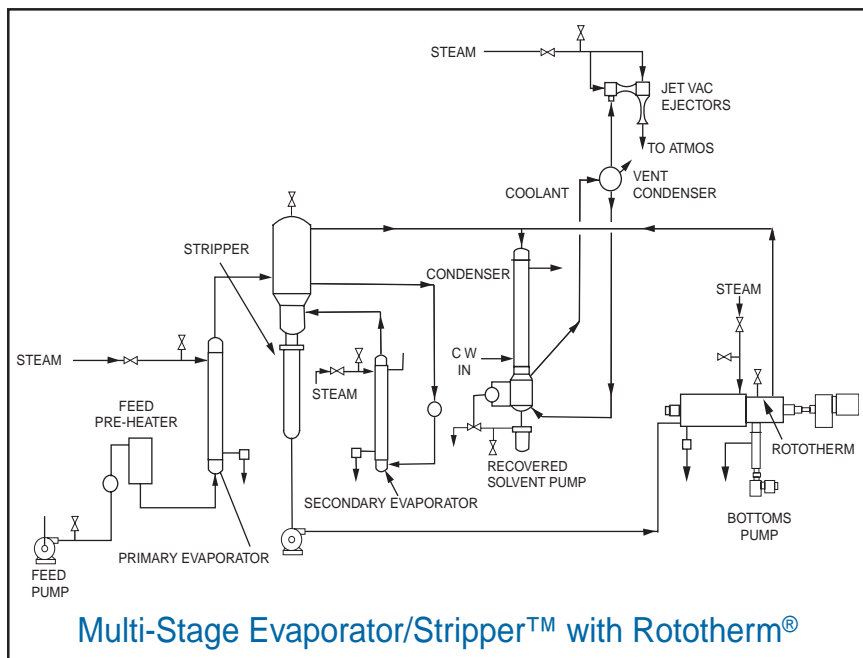


*65 years of chemical processing*

# Process Solutions Handbook



Multi-Stage Evaporator/Stripper™ with Rototherm®

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Artisan Industries Inc.

## Process Solutions Handbook

The Specialty Chemical Industry is dynamic and fast growing. Competition for product and performance has never been greater. For over sixty-five years Artisan has helped the chemical process industry pioneer benchmark technology platforms and unique solutions. Recognizing the increasing pace of commercial activity and the need for new ideas, Artisan has assembled this “Handbook” of proprietary solutions to share with our clients. We invite you to read further to learn how your manufacturing or product development can benefit from our years of challenging assignments.

Evaporator/Stripper is a trademark and , Jet-Vac, DUALFLO and Roto-therm are registered trademarks of Artisan Industries Inc.

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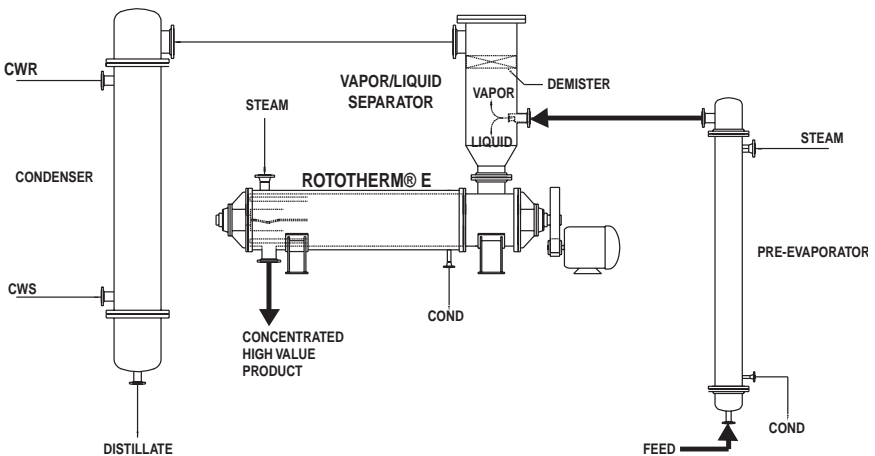


# Concentrating High Value Liquids or Slurries

**Problem:** Producing concentrates with little or no moisture from liquid or slurry feeds.

**Solution:** Artisan's mechanically agitated thin film processors concentrate heat sensitive, solids containing materials from very high moisture content in a single step, eliminating the need for costly, high maintenance filters, centrifuges, or spray type dryers.

**Example:** Concentrating an artificial sweetener containing water, alcohol, sugar, salt, and suspended solids, producing a concentrate with little or no moisture; coffee; tea; TDI; styrene, etc.

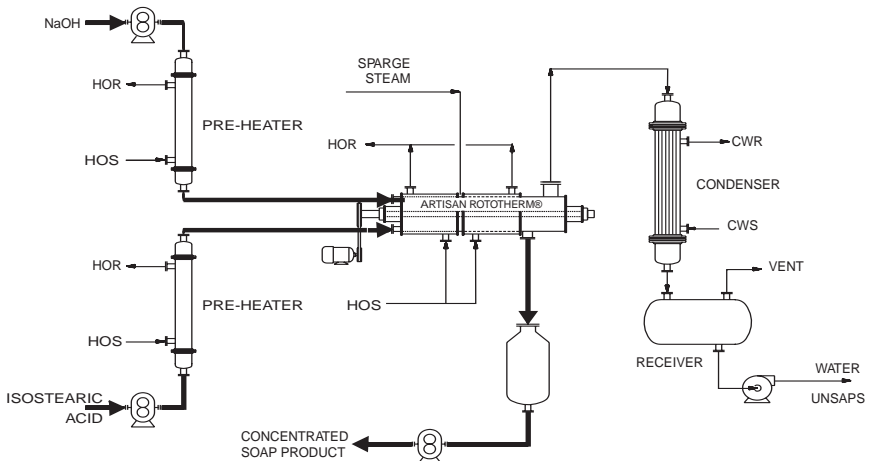


# Continuous Chemical Reactions With Product Concentration

**Problem:** Carrying out an equilibrium reaction while simultaneously concentrating the reaction product and discharging a solid or a slurry - in a single step.

**Solution:** Artisan two-zone mechanically agitated thin film processors are used to carry out continuous reactions. The co-current design, with two zone process section, permits reaction, concentrating the reaction products and discharging a heavy slurry or solid - all in a single step.

**Examples:** Single stage saponification of isostearic acid with sodium hydroxide, forming a stoichiometric soap, and steam stripping the “unsaps” from the soap, discharging a paste purified soap with an acid value of 195, and an unsap value  $<1$ ; also, esterification; acetylation; ketalization.

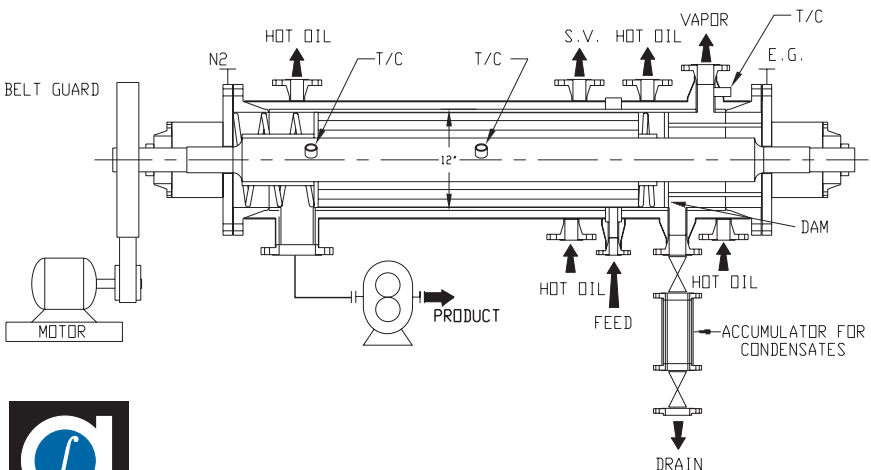


# Devolatilizing and Reacting Polymers

**Problem:** Efficiently removing solvents or monomers to low residual levels from shear and/or heat sensitive polymers. Typically, vent extruders accomplish this process task. However, extruders have limited heat transfer area per unit volume and rely on mechanical means, i.e. shear, to transfer energy into the material. In many instances, the high shear rates required lead to polymer degradation. In addition, the reduced area for vapor disengagement can lead to significant entrainment. Therefore, the initial solvent concentration in the feed is limited. To solve the vapor disengagement problem, equipment with smaller length to diameter ratios has been used. However, this technology is typically very inefficient at transferring heat and requires extended residence time for a given devolatilization, causing degradation in some heat sensitive polymers.

**Solution:** The Artisan horizontal thin film processor provides a unique combination of efficient heat transfer, short residence time and mechanical robustness to handle high viscosity polymers. The result is efficient processing of viscous materials without product degradation.

**Example:** Continuous polymerization reaction (finishing step), producing 2,000,000 CP polyester film.

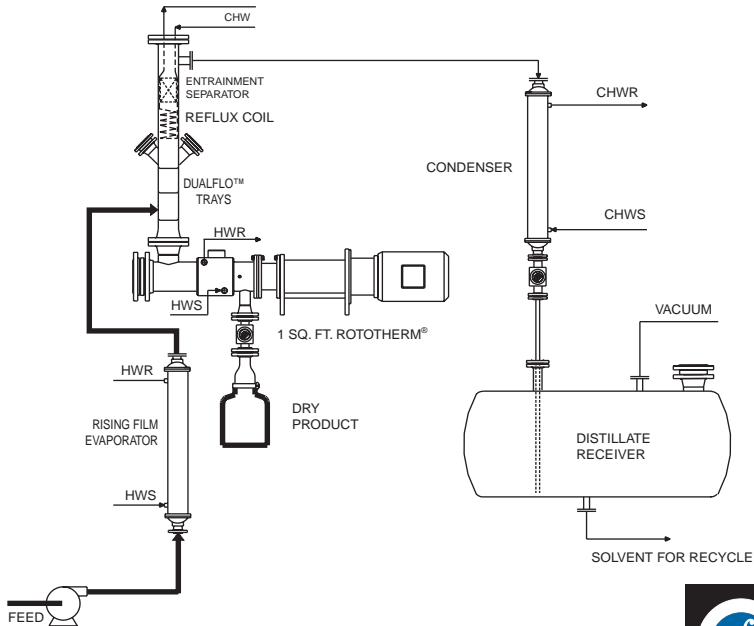


# Drying High Value Liquid Products

**Problem:** Producing free flowing powders with little or no moisture from liquid or slurry feed products.

**Solution:** Artisan's mechanically agitated thin film processors are widely used to produce free flowing powders from a variety of heat sensitive products. Rapid heat transfer, plug flow, and extremely short residence time make them the most versatile “single step” processors in the industry.

**Example:** Drying a heat sensitive “photographic dye” by evaporating the solvent from a 2% solution at reduced pressure. Pre-concentration in a rising film evaporator optimizes the size of the Rototherm®. A rectifying column, using the Artisan proprietary DUALFLO® Distillation trays, controls solids entrainment.

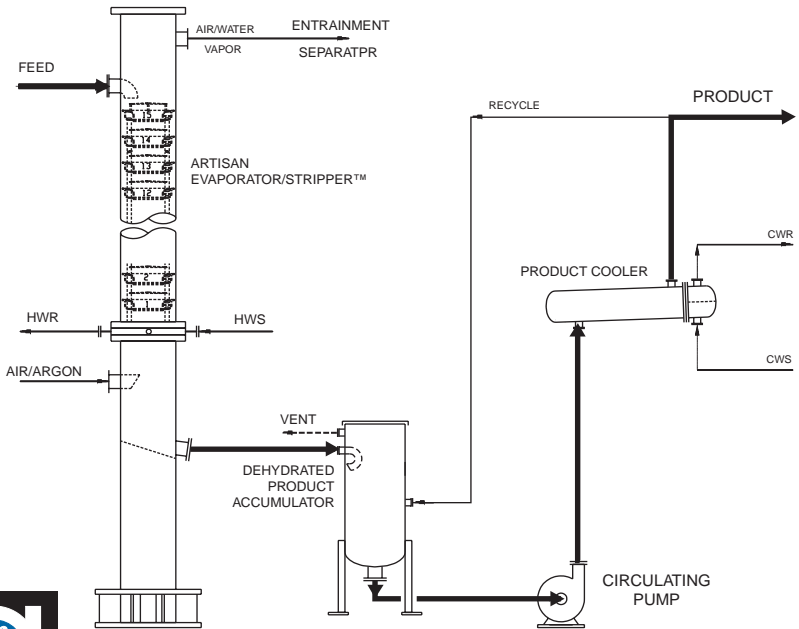


# Drying Explosive or Highly Flammable Organics

**Problem:** Continuous dewatering of organic peroxides or other highly flammable organics.

**Solution:** Artisan's patented falling film disc and tube Evaporator/Strippers™ are utilized to dry organic peroxides used as curing agents in polyester and vinyl chloride polymerization. Low holdup volume and “no crevice” design make it the safest method for continuous drying of these highly explosive or flammable organics.

**Example:** Drying methyl ethyl ketone peroxide from 5000 PPM to less than 200 PPM, by countercurrent stripping, using argon as stripping media.



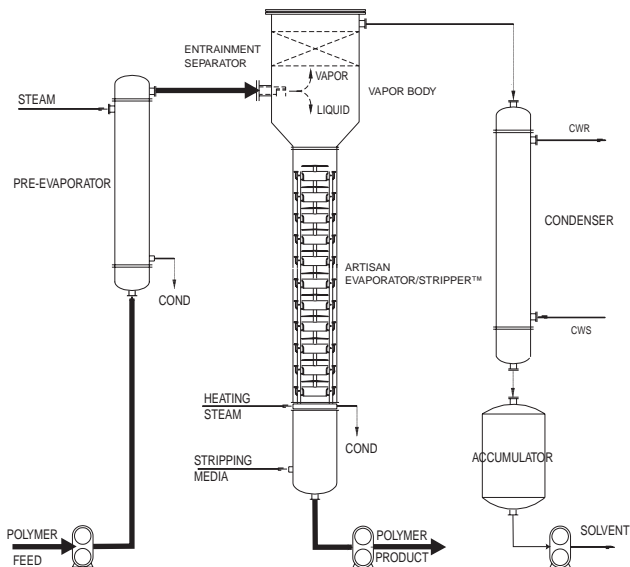
# Desolventizing Solution Polymers

**Problem:** Desolventizing and deodorizing solution polymers.

**Solution:** Artisan's patented falling film disc and tube Evaporator/Strippers™ can effectively desolventize and strip to low PPM levels a variety of solution polymers in a single pass, without product degradation. The Evaporator/Stripper's high mass transfer capability greatly reduces stripping steam. Product quality is enhanced since stripping is carried out as a thin film, in a single pass.

## Examples:

- Reducing toluene in a heat sensitive epoxy resin polymer from 50% to less than 200 PPM in a single pass.
- Continuous removal of residual styrene monomer from copolymer, from 3000 PPM to 8 PPM.
- Continuous removal of ethyl acetate from photographic dye dispersion.
- Continuous removal of high boiling volatiles from soap based latex emulsions.
- Continuous removal of vinyl chloride monomer from aqueous copolymer emulsion.

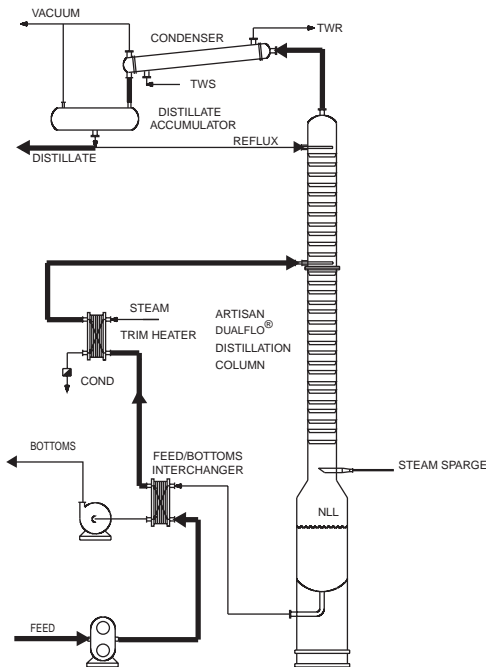


# Distillation of Solids Containing and Fouling Materials

**Problem:** Distillation of solids containing, foaming, and fouling aqueous and organic materials.

**Solution:** Artisan's proprietary DUALFLO® distillation trays demonstrate superior performance in severe applications where packing or conventional trays have failed. Compared to conventional trays, DUALFLO trays eliminate the need for overflow weirs and downcomers. DUALFLO trays use the entire column cross-section as active mass transfer surface, enabling higher throughput and eliminating dead spots. Simplicity of design and ease of cleaning greatly reduce both installation and operating costs.

**Examples:** Steam stripping VOC's from ground water, steam stripping emulsions and suspensions, distilling solvents from carbohydrates, and distilling solvents from aqueous salt solutions.



# Stripping Heat Sensitive Products

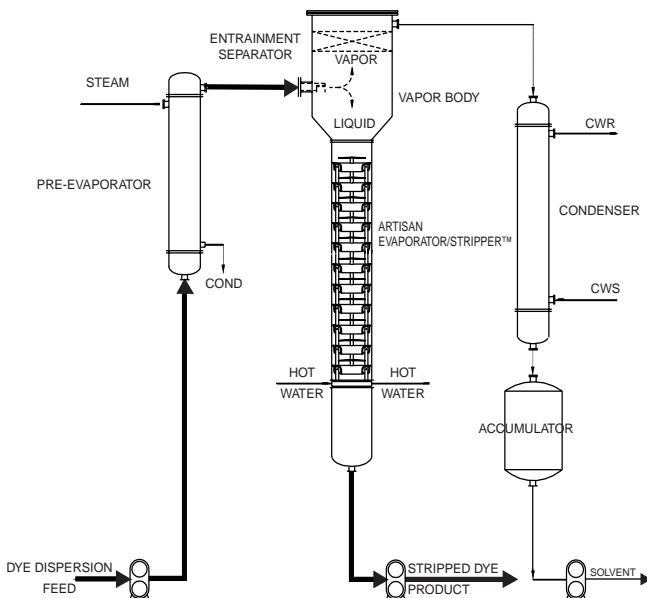
**Problem:** Stripping heat sensitive, viscous, or solids containing products, such as vitamins, fatty acids, plasticizers, phenolic resins, and pesticides.

**Solution:** Artisan proprietary falling film Evaporator/Strippers™ are widely used to strip solvents from heat sensitive and solids-containing high value products. Short residence time, high surface to volume ratio and extremely low pressure drop make it the most economical alternative to vertical wiped film evaporators (WFE). In most cases, one Artisan stripper can replace two or more WFE's.

## Examples:

Stripping:

- VOC's from latex emulsions to PPM level
- monomer from copolymer
- solvent from heat sensitive, unstable dye dispersions



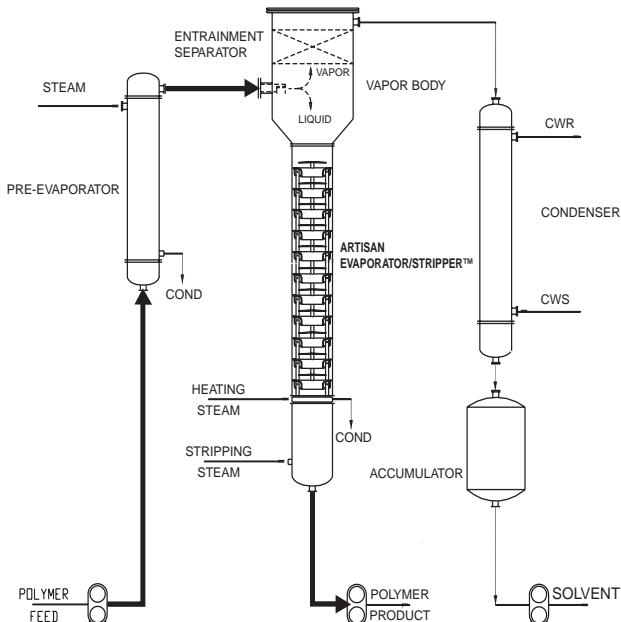
# Desolventizing Latex Emulsions

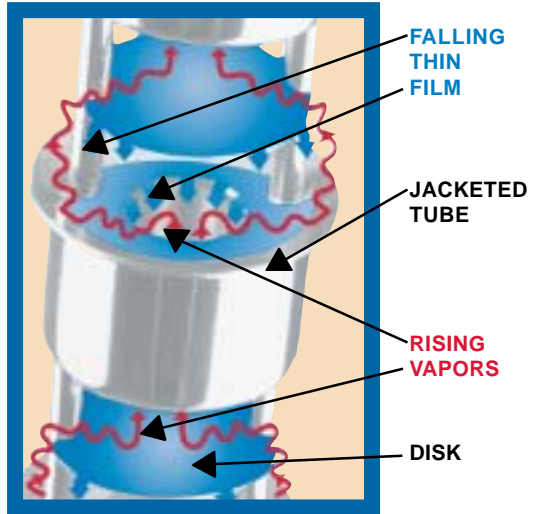
**Problem:** Desolventizing and stripping latex emulsions.

**Solution:** Artisan falling film disc and tube Evaporator/Strippers™ effectively desolventize and strip to low PPM level a variety of lattices—in a single step and without product degradation. The Evaporator/Stripper's high mass transfer capability greatly reduces steam consumption.

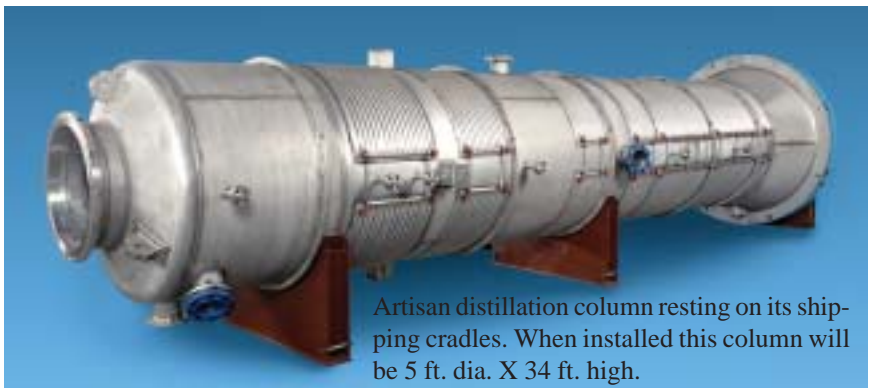
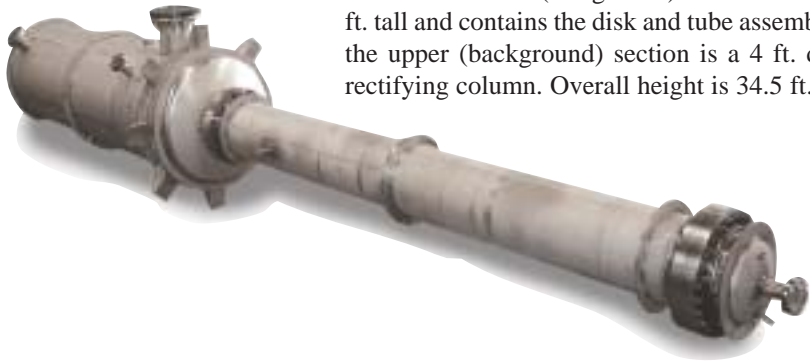
## Examples:

- Continuous removal of residual styrene monomer from copolymer.
- Continuous removal of ethyl acetate from photographic dye dispersion.
- Continuous removal of high boiling volatiles from soap based latex emulsions.
- Continuous removal of vinyl chloride monomer from aqueous copolymer emulsion.





Above: Liquid and vapor flows around Evaporator/Stripper™ disks and tubes. Left: full scale disk and tube assembly. Below: Evaporator/Stripper column awaiting shipment. The lower section (foreground) is 20" dia. X 18 ft. tall and contains the disk and tube assembly; the upper (background) section is a 4 ft. dia. rectifying column. Overall height is 34.5 ft.



Artisan distillation column resting on its shipping cradles. When installed this column will be 5 ft. dia. X 34 ft. high.



Complete skid-mounted systems are an Artisan specialty.



2200 ton Jet-Vac® vacuum chiller



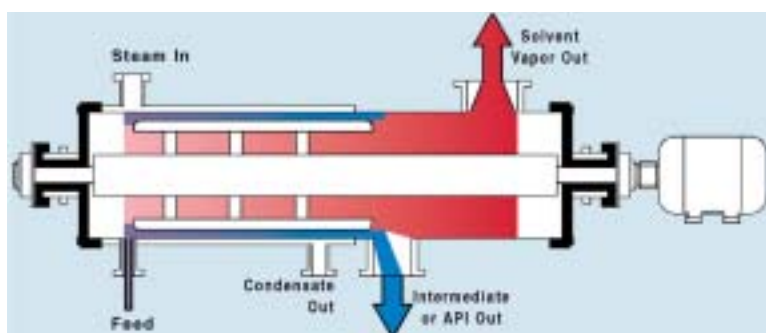
20 sq. ft. Rotothem® with Jet-Vac® hybrid vacuum system. This Rotothem concentrates and produces free-flowing powder for the food industry.



200 sq. ft. Rotothem® Horizontal Thin Film Evaporator



Installed in a cGMP facility, this Rototherm is designed to purify API, intermediates or fine chemicals.



Liquid and vapor flows in the Artisan Rototherm®.



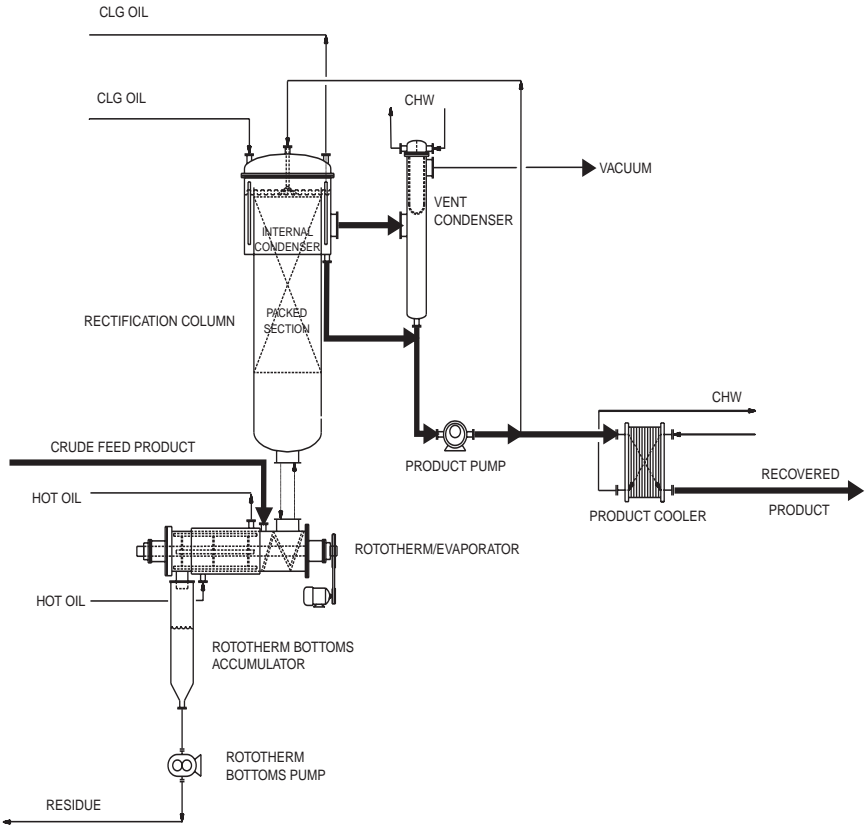
## Recovering heat sensitive API's, intermediates or fine chemicals from solvent

**Problem:** Many compounds are produced in dilute solutions of solvent or are purified in mobile solvent streams, as in the case of chromatography. Heat sensitive, biologically active or intermediate structures within solvent laden, foaming, and/or fouling solutions are challenging mixtures for the recovery of valuable pharmaceutical or fine chemical molecules. Heat sensitivity presents a critical problem because elevated temperatures and exposure time must be controlled to preserve molecular integrity. Solutions with difficult compositions may foul heat transfer surfaces or form disruptive foams that inhibit the process. If heat transfer surfaces foul, throughput is reduced.

**Solution:** The Rototherm horizontal processor overcomes the problems of fouling, foaming, heat sensitivity, solvent content, etc. It allows concentrating and isolating the important ingredient quickly, often in seconds, in a single step. The process can be turned down to accommodate variable processing rates. The process is scalable to any capacity, and active compound efficacy can be demonstrated easily at small volumes and very low concentrations. The combination of vacuum operation and short residence time prevents product degradation. The unique rotor in the Rototherm processor allows for efficient self-cleaning of the internal heat transfer surfaces. The highly turbulent thin film at the heated surface enables heat flux rates of 10,000 to 50,000 BTU/hr-ft<sup>2</sup>. Many feed and processing strategies can be employed to optimize the operation.

**Applications:** Active Pharmaceutical Ingredients (API), specialty chemical compounds.





# Reboiler Strategies for Heat Sensitive Products

**Problem:** Distillation of heat sensitive, fouling/foaming, viscous or solids containing “high value” products presents several operating challenges:

- **LOW OPERATING PRESSURE:** Generally, heat sensitive materials must be processed at very low absolute pressures, such as 1 mbar, to reduce operating temperatures.
- **LOW RESIDENCE TIME:** These products require extremely low temperature and time exposure during processing.
- **HIGH YIELD:** High market value (anywhere from \$10 or \$15 per kilo, and up to \$1,000 or more) means maximum recovery is the key to profitability.
- **HIGH PURITY:** Purity, typically specified to the second or the third decimal point (99.995%), is perhaps the most critical measure of success in the recovery of high value products.
- **LOW COLOR:** Another critical quality parameter is color, which is generally required to be “water white”.

**Solution:** Use Artisan’s proprietary “thin film evaporators” as column reboilers, in lieu of conventional technologies, such as thermosyphons, forced circulation, or even falling film evaporators. A highly turbulent film, extremely short residence time, narrow residence time distribution and rapid surface renewal make the Rototherm the most versatile processor in the industry.

**Example:** Purification of antioxidants used as preservatives in variety of products including soaps, oils, food products (such as vitamins), plastics, rubber, and gasoline.



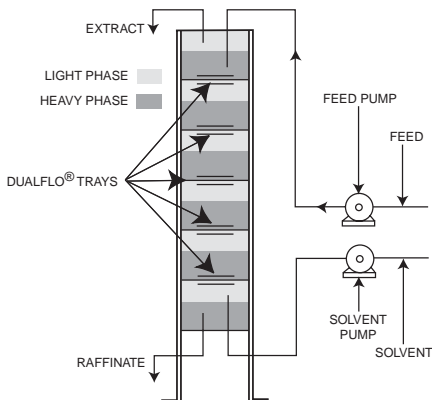
# Recovering Selected Organics by Extraction

**Problem:** Recovering high purity organics from azeotropic or close boiling aqueous solutions is uneconomical using conventional distillation.

**Solution:** Use a closed-loop, cost effective, energy efficient Artisan controlled cycle liquid-liquid extraction system to extract the desired organic using a solvent with a higher affinity for the organic than water. Then use conventional distillation to separate the solvent from the organic. Purify the raffinate water in a steam stripping column to produce disposable water. Distillation overheads are recycled back to the recovery system, creating a process with no waste stream.

## Examples:

- Recovering methyl ethyl ketone from water, using toluene to extract the methyl ethyl ketone. Dehydrate the extract by simple distillation, producing a methyl ethyl ketone/toluene stream to be separated by conventional distillation. This produces both high purity methyl ethyl ketone, as well as toluene for recycle to the extractor. Purify the raffinate water in a steam stripping column, producing disposable water.
- Recovering acetic acid from a weak aqueous solution, using ethyl acetate to extract the acid. By distilling the ethyl acetate rather than water, you cut energy consumption by roughly 85%.

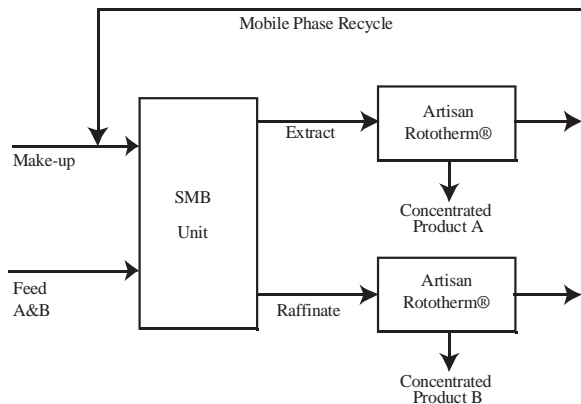


# Managing Solvents in Chromatographic Separations

**Problem:** Separating compounds using single column prep LPLC, HPLC, or multi-column SMB generates significant quantities of solvent from which the separated compounds must be quickly and economically recovered. Traditional batch glass rotary evaporators become unreasonably large and risky at the sizes necessary to handle the solvent volumes associated with batch prep chromatography and continuous SMB.

**Solution:** Artisan mechanically agitated thin film evaporators are being used instead of glass rotary evaporators. The short residence time is ideal for recovering heat sensitive products from the solvents. The low liquid hold up minimizes safety risks due to the short exposure time of the solvent at elevated temperatures. Finally, the wide range of capacities, and extremely high turn down and evaporation capability provide the processing flexibility required in typical laboratory or pilot environments.

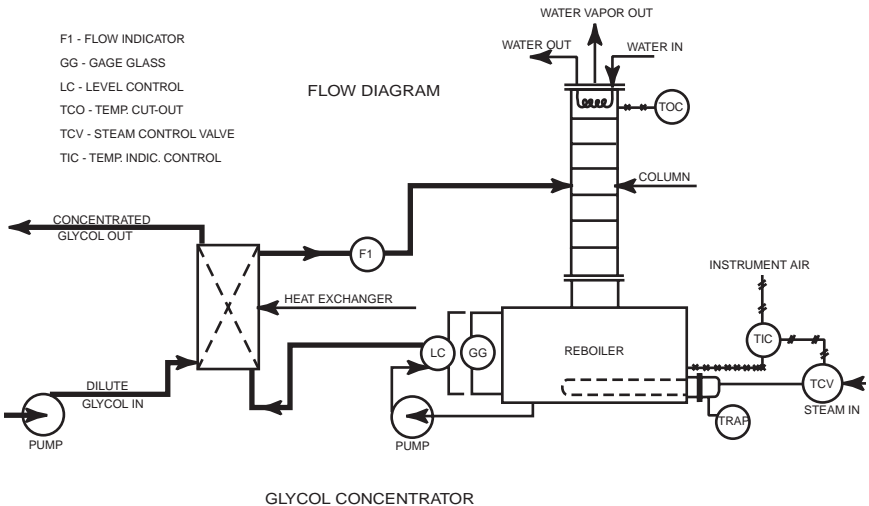
**Examples:** Two Artisan mechanically agitated thin film evaporators are in operation for product recovery and solvent recycling at an SMB contract manufacturing facility. The evaporators are validated for cGMP operation.



# Purifying Glycols

**Problem:** Recovering ethylene or propylene glycol (used as antifreeze to disperse and remove ice forming on condensers or for deicing aircraft) that is contaminated with floating particles such as dust, salt, sand, etc.

**Solution:** Use Artisan DUALFLO® distillation trays. For over forty years, companies have recovered high purity reusable glycol using these proprietary trays. The water vapor stream is glycol free (20 PPM or less) and safe for discharge to the atmosphere. DUALFLO trays do not use down comers or overflow weirs and, therefore, stay clean and maintenance free longer than conventional trays.



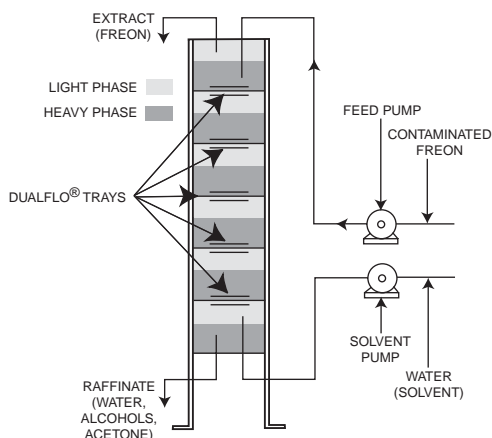
# Recovering and Purifying Freon by Extraction

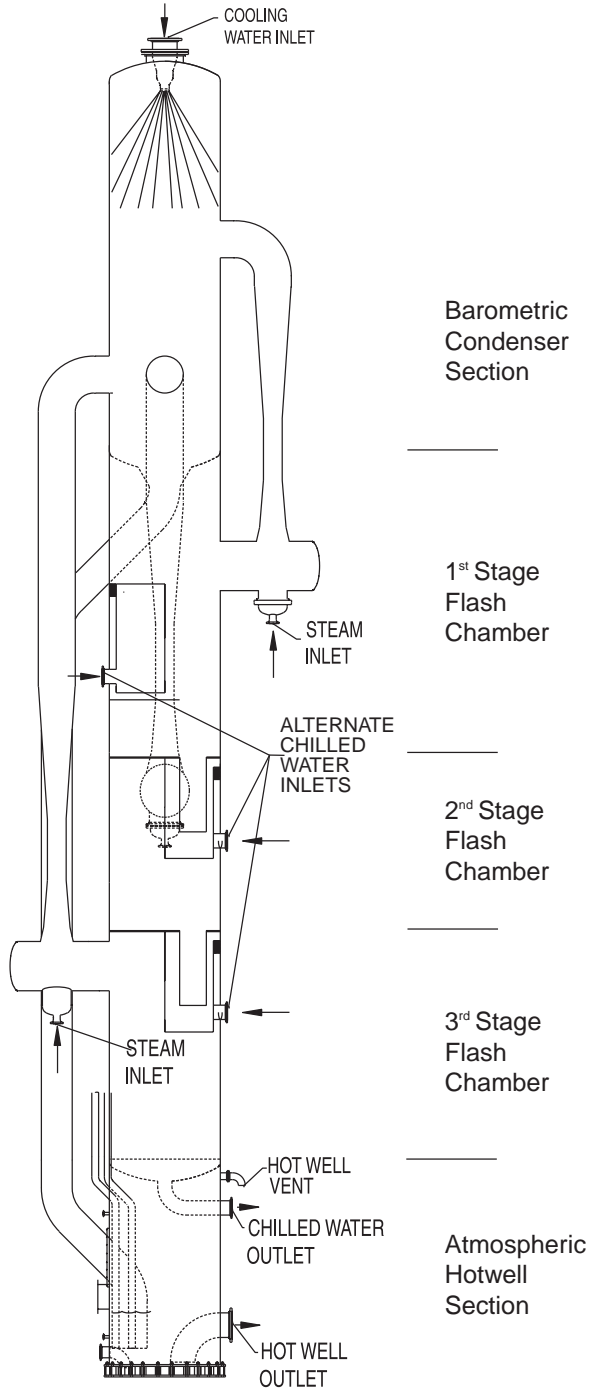
**Problem:** Recovering high purity Freon from a mixture of water-soluble contaminants such as, isopropyl alcohol, acetone and 1,4 dioxane would require complex multistep distillation because they have similar boiling points.

**Solution:** Use an Artisan Liquid/Liquid extractor to extract the contaminants using water as the solvent. In this separation technique, the mixture countercurrently contacts a stream of water in a DUALFLO<sup>®</sup> column. The alcohols and acetone are more soluble in water and become part of that stream. Polish the Freon, now essentially water free, with a desiccant to less than 10 PPM final moisture content. Steam strip the contaminated water and return it to the extractor as solvent.

## Examples:

- Continuous extraction of theobromine and caffeine from a water solution with methylene chloride as solvent, using the Artisan DUALFLO<sup>®</sup> tray extraction column.
- Single step, continuous purification of triphenyl phosphate (TPP), by simultaneous caustic neutralization, and water washing using the Artisan DUALFLO<sup>®</sup> tray extraction column.





## Chilled Water: Alternative to Refrigeration

### Problems:

- a) Chilled water is required in large quantities, or
- b) a process fluid needs cooling.

### Considerations:

- the potential danger and expense associated with mechanical refrigerants (CFCs).
- the high maintenance, large space (real estate) requirements, and high operating cost (KwH) associated with mechanical chillers.
- a cooling tower that can not deliver a consistently low fluid temperature.
- ground or surface water sources not viable choices.

**Solution:** The Jet-Vac® chiller system produces chilled and/or deaerated water without refrigeration, and has the following advantages:

- no refrigerants
- no moving parts
- low operational cost
- unlimited quantities of chilled water
- small footprint

The principle of the evaporative cooling process is applied to cooling water or directly to the process fluid to achieve a low temperature and/or deaeration. A steam source at the site furnishes the motive force. A wide range of temperature differences or flows is possible.

### Examples:

Chilled Water			Cooling	Tower Size	Steam		Cooling Water	
GPM	°F out	°F Rtn	Tonnage	(inch Dia x Ft tall)	psig	#/HR	GPM	°F in
6000	74	84	2500	126x120	100	27,000	11,000	85
4000	68	81	2200	100x110	25	27,000	6,500	87
2000	75	85	835	66x62	180	11,000	4,200	90
1200	60	90	1500	78x75	15	35,000	7,000	90
200	40	59	160	42x50	135	3800	700	80
125	50	68	100	30x50	70	2500	600	90



# Natural Gas Recompression: Recovering Lost Gas

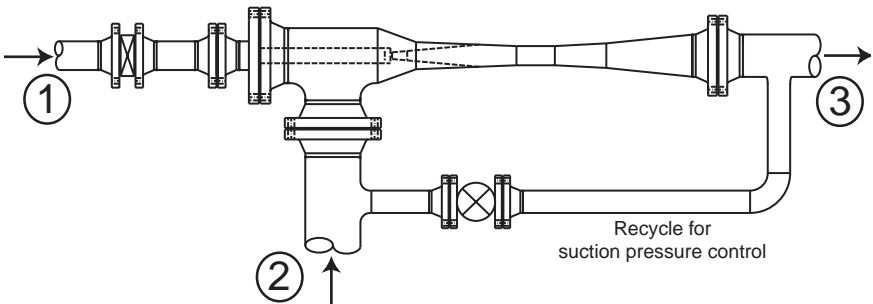
**Problem:** Recovering low quality gas or fluid that might otherwise be wasted or lost to the environment.

The boil off gas from LNG storage tanks is captured in weighted low pressure ground tanks but this storage strategy presents these problems:

- maintaining the seals is very expensive
- real estate requirement is enormous
- local regulators consider the situation unsightly and dangerous

**Solution:** The recompression concept incorporates a higher pressure stream of gas (motive gas) with the waste or low pressure stream, and then creates a third combined stream of intermediate pressure. The advantages of the process solution are:

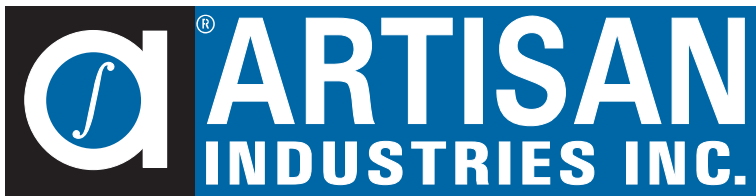
- no moving parts (non-mechanical)
- no electric drives
- recaptures low pressure vapor and compresses it to a usable level



## Example:

①	Motive Gas:	29,000 lb/hr	at	125 psig
②	Suction Gas:	10,000 lb/hr	at	0 psig
③	Discharge Gas:	39,000 lb/hr	at	15 psig





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