

# LABTEX™

The UK's Laboratory Equipment Experts



## Distillation and Evaporation

Pope Scientific has been designing and manufacturing distillation and filtration equipment for Labs, Pilot Plants, & Production Processing worldwide for over 50 years.

Pope's short path/wiped film stills, evaporators, molecular stills and fractional distillation columns can be found working in R&D laboratories, universities, small & large companies throughout the world.

[www.labtex.co.uk](http://www.labtex.co.uk)

# Thin Film Distillation and Wiped Film Evaporators

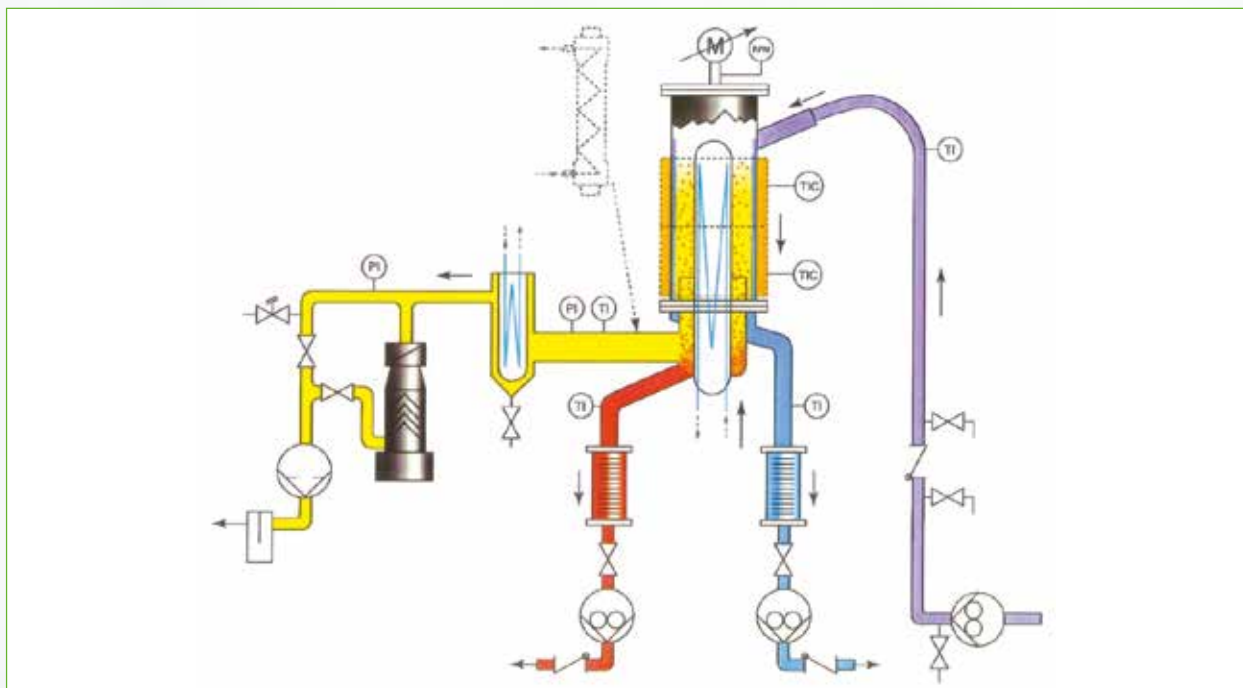
Pope Scientific Short Path/Wiped-Film Stills (WFS) and Wiped-Film Evaporators (WFE) successfully separate volatile from less volatile components for Oils, Fats, Chemicals, Polymers, Nutraceuticals and Fragrances etc. with a gentle process utilizing the thin-film wiping action of feed liquid through a heated cylindrical vacuum chamber with high vacuum (i.e. vacuum distillation/evaporation).

## Keys to the superiority of this process include:

- Short residence time of the feed liquid
- Significantly lowered temperature due to high vacuum capability, and
- Optimal efficiency in mass and heat transfer

The brief (seconds) exposure of feed liquid to heated walls is due in part to the slotted wiper design which forces the liquid downward with strict control of residence time, film thickness, and flow characteristics.

Efficient thermal separation with minimum product decomposition and maximum product quality result when using the Wiped Film-Short Path process for distillation. The Wiped Film process offers far superior performance to flash evaporators, falling film stills, rotary evaporators and similar equipment in any processing application where heat-sensitivity is a factor.

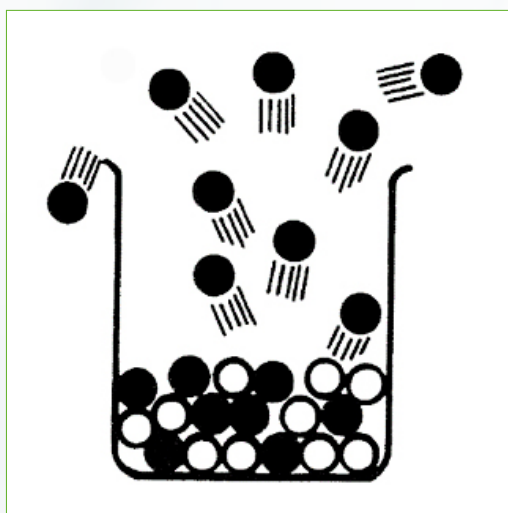


# The Molecular Background

**Wiped-Film still technology takes advantage of the fact that each chemical substance has a characteristic vapour pressure. It is this relative difference in vapour pressures which dictates how easily a complex compound can be separated into its constituent components.**

Since the molecules of all matter are in constant motion in varying degrees, depending upon the chemical composition of that matter and the temperature and pressure applied to it, molecules near the surface have a tendency to escape into the surrounding atmosphere. As temperature increases and pressure decreases, this escaping tendency usually increases and the substance is said to vaporize.

The force generated by these escaping molecules is referred to as the vapour pressure of that material at a particular temperature and pressure. It is the relative difference in vapour pressure of substances which dictates how easily a complex compound can be separated into its constituent compounds.

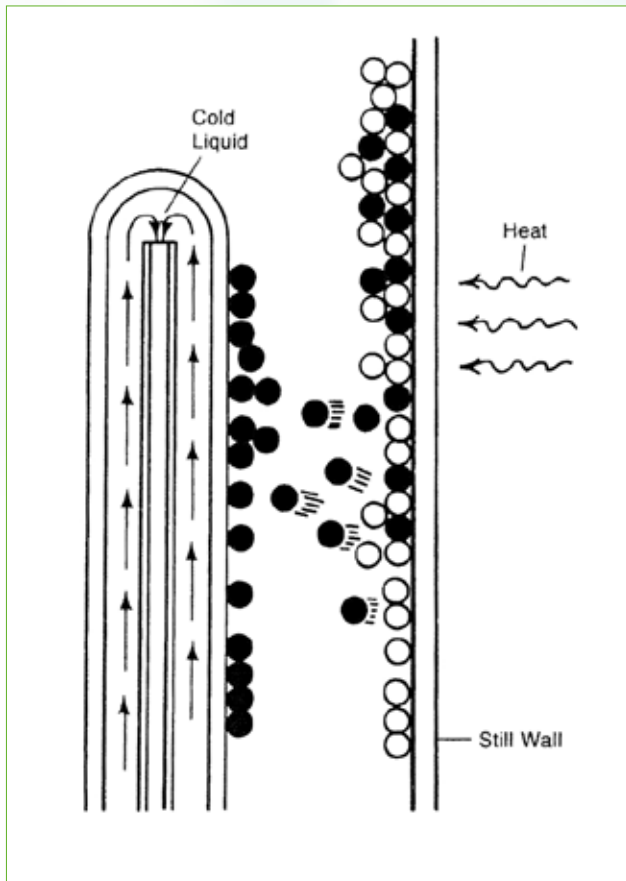


*Molecules escaping from solution.*

# Brief Operating Description

The Pope Wiped-Film Still consists of a heated body into which a fluid system requiring some degree of separation is continuously fed. The fluid is spread into a thin film by a rotating wiper blade assembly driven at a predetermined speed. The film, while being forced into turbulent flow by the wiper blades, progresses down the inside body wall aided by gravity and the slots in the wiper blades.

During the course of flow through the heater system, some degree of evaporation takes place depending upon the characteristics of the feed material and the inside wall temperature, in addition to the system pressure. The non-evaporated fluid forming the bottom product flows out of the system continuously while the vapour is condensed either inside or outside the system depending on the type of design.

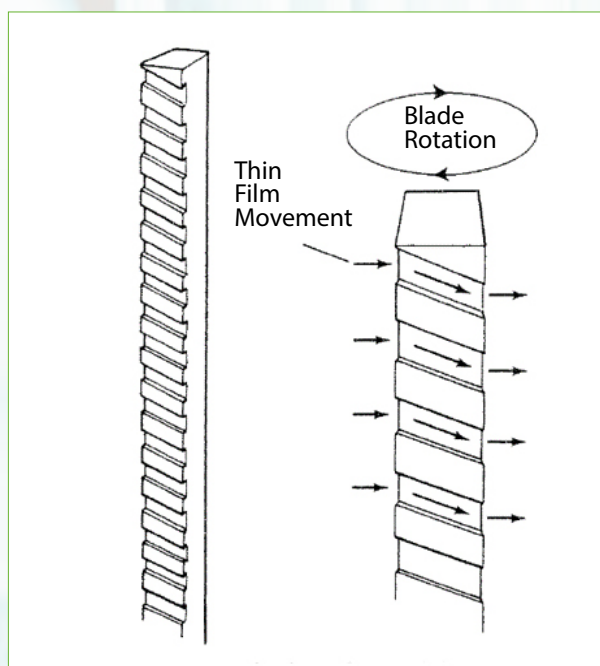


*Basic evaporation and condensation.*

# Why Thin Films?

## Thin films are created in Pope Wiped-Film Stills for a variety of reasons:

1. Turbulence created by a rapidly moving wiper or controlled clearance blade greatly assists in heat transmission, thereby lowering the temperature required on the inside evaporator wall for a given system pressure.
2. A maximum surface area per unit volume of flow is generated facilitating rapid evaporation.
3. The liquid exposure time to the elevated wall temperature can be controlled within seconds or less. This minimizes product degradation of heat sensitive materials by controlling the wiper assembly speed.
4. High viscosity materials can be transported through the system for distillation or solvent stripping.
5. Pope slotted wiper blades promote plug flow /with little back mixing. This minimizes dwell time distribution ensuring that material flowing through the system has a uniform exposure to process conditions.



*Wiper blade moves each plug of material downward.*

# The Pope Dimension of Excellence

**Pope adds superior design and construction to the basic wiped-film still process. This has enabled Pope stills to sustain pressures down to one micron for either solvent stripping or molecular distillation.**

Pope variable speed control of the wipers coupled with the ability to reverse rotation, allows an extremely wide variation of hold- up time for the process fluids in the evaporator body. Pope supplies either Teflon or carbon wiper blades with carbon preferred on applications with temperatures over 200 degrees Centigrade.

When increased condensing capacity of a non- corrosive distillate is required, the conventional glass “cold finger” can be replaced by a stainless-steel condenser presenting a unique combination of Pope process capabilities. It is the ability of Pope craftsmen to work in both glass and stainless steel and combinations of both that provides a unique dimension of excellence — a combination of process variables not available elsewhere. Pope Scientific offers you flexibility!

In those instances when corrosiveness to stainless steel is a problem, Hastelloy, Titanium, C-20, other alloys, or glass can be offered as the preferred construction materials. To generate sound research and development data for molecular distillation or solvent stripping process, variables must be rigorously controlled — i.e. feed rate, wiper revolution rate, still body temperature and pressure. Pope Scientific provides the capability to control each of these variables to the degree required for efficient and repeatable processing.

## Typical Applications

The thin film evaporator/molecular still technique has been successfully utilized since the 1940's. It remains a primary means of separation for many applications, including:

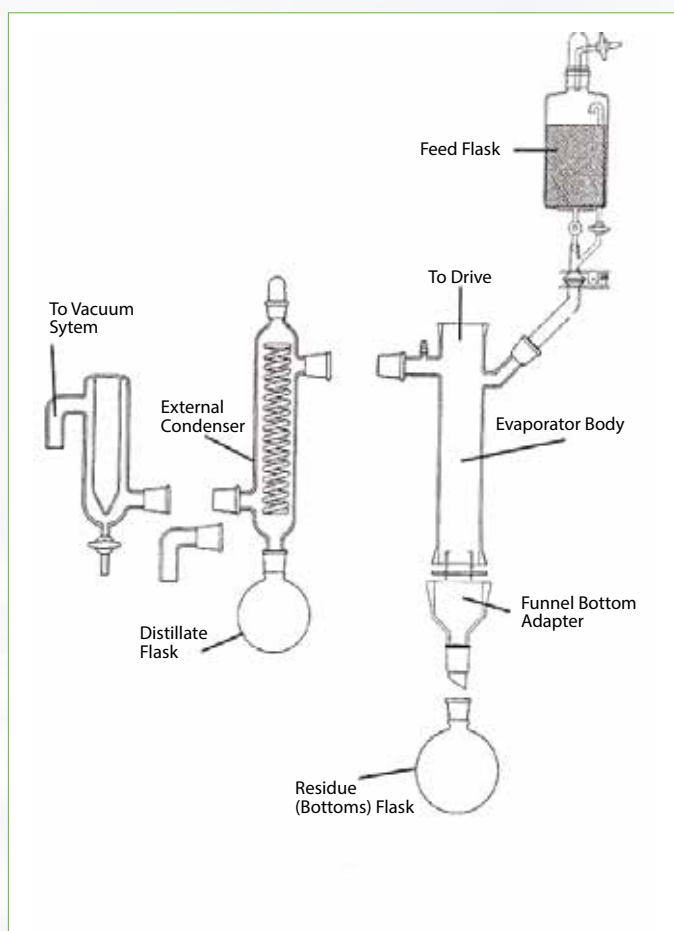
- Solvent stripping
- Stripping monomer from polymer
- Stripping free fatty acids from fats and oils
- Distilling fats and oils
- Concentrating or distilling heat –sensitive pharmaceuticals, nutraceuticals and biomaterials
- Distillation of polymers
- Distillation of petroleum fractions
- Reactives from solid catalysts
- Concentration of fruit juices
- Isolation of aromatic compounds
- Deodorization of oils
- Removal of colours
- Separations of waxes or silicones
- Foods and flavours purifications



# Basic System Configurations

Pope Wiped-Film Stills are currently offered in three basic models with differing evaporating surface sizes:

1. 2" model with a 323 sq. cm. (.35 sq. ft.) evaporator surface.
2. 4" model with a 1,097 sq. cm. (1.18 sq. ft.) evaporator surface.
3. 6" model with a 2,194 sq. cm. (2.36 sq. ft.) evaporator surface.



**Figure 1:** Pope Concentration/Evaporation unit with necessary components.

Model size is based on the diameter of the evaporating chamber. Still components are normally made from borosilicate glass except for the drive system (usually 316 stainless steel) and wiper blades (Teflon™ or ultra-pure high temperature carbon).

Other materials are furnished on special order– such as stainless steel or quartz in place of glass and other metals substituted for the drive.

Pope stills are available in a broad range of variations, to more efficiently accomplish the following different types of separation:

## Concentration/Evaporation

In the case of concentration/evaporation, the desired final product is usually the residue (bottoms), not the distillate and water is often the substance removed. Therefore, broader cuts can be made to remove the unwanted material as distillate. The throughput is also usually larger than other types of separation.

Because of this, the efficiency of the operation is improved by the following factors:

1. Larger feed and receiver flasks.
2. Extra condensing power, as supplied by Pope external condensers, or stainless steel internal condensers, rather than the glass internal condenser.
3. Higher heating capacity, as affected by metal band heaters, heat transfer fluids or stainless steel bodies.

A basic system for concentration/evaporation will include the wiper drive, wiper blades, evaporator body with internal condenser, feed and degasser flask, receiver flasks, external condenser and receiver flask, adapter from condenser to vacuum and the vacuum system. The type of heating system and temperature control will depend upon such factors as maximum body temperature, desired throughput, etc.

“Temperature Control Systems.” Additional components, such as a liquid nitrogen cold trap, various instruments, etc., can also be supplied.

## Molecular Distillation

In molecular distillation, the desired final product is usually the distillate. Pope Still bodies are dimensionally designed for more efficient molecular distillation. The condenser is situated inside the evaporator, with its condensing surface sufficiently close to the evaporating surface so as to minimize collisions of evaporated molecules on their path from evaporator to the condenser.

Molecular distillation is improved by using:

1. High vacuum systems
2. More precise temperature control

A basic system for molecular distillation will include the wiper drive, wiper blades, evaporator body with internal condenser, feed and degasser flask, receiver flasks, liquid nitrogen cold trap and the vacuum system. The type of heating system and temperature control utilized will depend upon such factors as maximum body temperature, desired throughput etc.

Additional components, such as instruments, can also be added.





# Solvent Stripping

**Solvent stripping is essentially the same type of separation as concentration/evaporation. The desired product is usually the residue (bottoms).**

The distillate (the removed solvent) is generally sufficiently pure enough to be reused. Because the latent heat of vaporization for solvents is typically, 1/4 to 1/6 that of water (which is usually removed in the evaporation/concentration mode), throughputs are considerably higher. Feed pumps are often used to pump the feedstock out of large feed vessels.

Thus, a run of many hours can be performed without any operator assistance necessary on Pope automated systems. Note that the largest throughputs are obtained with the stainless bodies using an external condenser. A representative system for solvent stripping will include the wiper drive, wiper blades, evaporator body with stainless steel internal condenser, large feed flask with feed pump, receiver flasks sized to accommodate desired throughput, external condenser with receiver flask, adapter from external condenser to vacuum system and the vacuum system. As in all cases, the type of heating system and temperature control will depend upon such factors as maximum body temperature, desired throughput, etc.



2" Bench top evaporator



Two stage 9" and 12" WFS plant



4" WFS with telescopic stand



4" Molecular still with external condenser



6" Hastelloy jacketed WFE pilot plant



6" glass WFS

# Application questionnaire

## APPLICATIONS QUESTIONNAIRE FOR MOLECULAR STILLs, EVAPORATORS, FRACTIONAL STILLs

Date:	Phone:
Name:	Fax:
Title:	Email:
Company:	
Address:	
	Postcode:

The following data is useful for properly sizing equipment and establishing process parameters to accomplish a desired separation. All data will be treated confidentially.

### I. FEED MATERIAL COMPOSITION & PROPERTIES

Please fill in as much of the information below as possible. It is essential that all components are listed and that the weight percentages total 100%, even if estimated. All provided boiling point and vapor pressure data, and at least some information on viscosity is extremely helpful.

Components (List All)	% by Wt in Feed (Total = 100%)	BP °F/°C (760 mm Hg)	MP °F/°C (760 mm Hg)	MW
1.				
2.				
3.				
4.				

### II. DESIRED PRODUCT PURITY

Which component(s) is the important end product? \_\_\_\_\_

Use the table overleaf to indicate desired goal (or ideal), and minimum acceptable final purities or compositions.



Components	Goal % Wt in Distillate	Acceptable % Wt in Distillate	Goal % Wt in Residue	Acceptable % Wt in residue
1.				
2.				
3.				
4.				

Further notes regarding end product purity: \_\_\_\_\_

### III. DESIRED PRODUCTION RATES (Indicate kg/hr. or gal/hr etc)

**A.** Feed : \_\_\_\_\_ **B.** Distillate : \_\_\_\_\_ **C.** Residue : \_\_\_\_\_

If required production rates are not yet known, please provide estimate of batch sizes (from a reactor, or other operation), and frequency of batches. [Examples: 5000 kg/wk, 6 gal/day, 200 metric tons/month, etc.]

### IV. ADDITIONAL INFORMATION (Add extra sheets, if necessary)

**A.** Vapor Pressure/Temperature Data (mm Hg. @ °C) \_\_\_\_\_

**B.** Viscosity/Temperature Data (cP @ °C) \_\_\_\_\_

**C.** Misc. Physical Information (Degradation Limits, Sublimation, Foaming, etc)

\_\_\_\_\_

**D.** Materials of Compatibility Information:

\_\_\_\_\_

**E.** Safety Information (Flammability, Flash Point, Toxicity etc.):

\_\_\_\_\_

**F.** Any other known information would be helpful:

\_\_\_\_\_



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ARE LEADERS IN THEIR  
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Thermoregulation



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Precision Balances



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(eg PTFE, PFA) Labware



Vacuum Pumps  
and Networks



Overhead and  
Magnetic Stirrers



Magnetic Stirrers, Overhead Stirrers,  
Dispersers and Rotary Evaporators



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