

FRACTIONAL DISTILLATION SYSTEMS

Standardized for Purification, Fractionation and Solvent Recovery

For pilot plants and small scale production

96
92
88
84
100
104
104
108

n - Heptane
98.4°C

Toluene
110.8°C

$$\frac{\dot{m}_A}{\dot{m}_B} = \frac{(p - n_s p_B) M_A}{n_s p_B M_B} = \frac{M_A}{n_s M_B} \left(\frac{p}{p_B} - n_s \right)$$

$$\frac{dx_{1.0}}{dt} = \left[\frac{L_0 + D + \frac{dM_0}{dt}}{M_0} \right] x_{1.0}$$

$$V_1 = D(R + 1) + \frac{M_0}{dt}$$



Solution Driven.

Pope Scientific is Now Offering...

Mid-Sized, Standardized Fractional Distillation Systems for Purification, Fractionation and Solvent Recovery.

Pope's new state-of-the-art line of mid-sized fractional distillation systems has been created to fill the gap between small laboratory bench scale units and large process systems. The 1 - 100 gal/hr throughput capacity is ideal for pilot plants and for applications requiring small production runs. These exceptional systems incorporate precision design and fabrication with meticulous finishing to rigorous standards. This makes them particularly suitable for critical applications such as fine chemicals, pharmaceuticals, foods, flavors, cosmetics, extracts, etc.

By standardizing our components, we have facilitated component selection and equipment upgrading and reconfiguration. Installation and startup are simplified. Plus, all our equipment can be made available in custom variations to fit your specialized requirements.

Pope Fractional Distillation Systems are available :

- for batch or continuous feed applications
- in 1 - 100 gal/hr range of throughput capacities
- with 2 - 12" column diameters, in standard flanged sections
- in glass or 316-L stainless steel (ceramic, plastics and alloys also available)
- with a wide variety of column packings and internals (Pro-Pak® protruded metal is our standard packing)
- as turnkey systems, partial systems and individual components

A rough estimation of size of column and energy required to achieve a desired separation of liquids can be obtained by referring to the tables on page 7* of this brochure. Column capacity varies with the column diameter, the relative volatility of the liquids being separated, the number of stages (theoretical plates) required for desired purity and by the column packing type and internals. Our system engineers can help you determine the system configuration that best suits your needs.

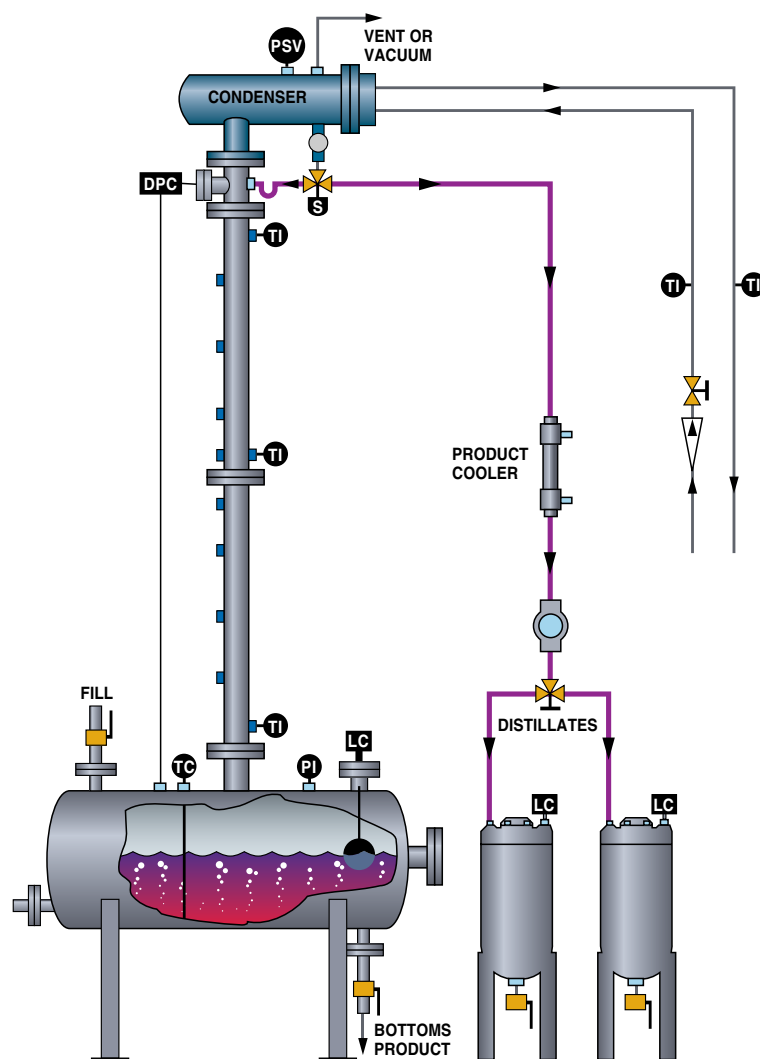
Pope Scientific offers complete product support in the form of training, technical support, parts and repair, and for large systems, turnkey installation.



* Note: This information has been condensed and simplified.

Batch Mode

When relatively small quantities of liquid are to be processed, when an extremely high level of purity is required or when more than 2 solvents need to be separated, batch mode is preferable.



Typical Systems Include:

- Boiling vessel with fill and drain valves, sight glass, sensors for liquid level, pressure and temperature, and extra ports.
- Choice of heating options (see specifications section).
- Column section(s), pre-loaded with packing and required internals.
- High efficiency condenser with cooling water lines, metering valve and flow meter.
- 3-way automatic reflux valve.
- Distillate product cooler and sight glass.
- Receiving vessels for cut fractions (discharge pumps optional).
- 3-way receiver selector valve (automated with Model 3000 Batch Distillation Controller).
- Pope Model 3000 Batch Distillation Controller.
- Skid platform and support structure for system.
- Insulation.

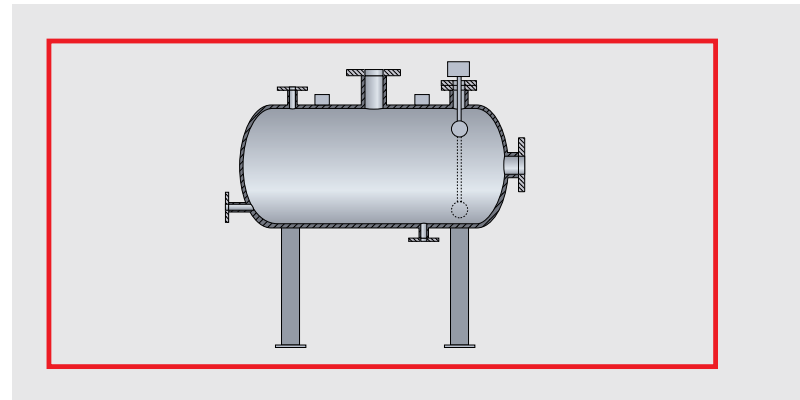
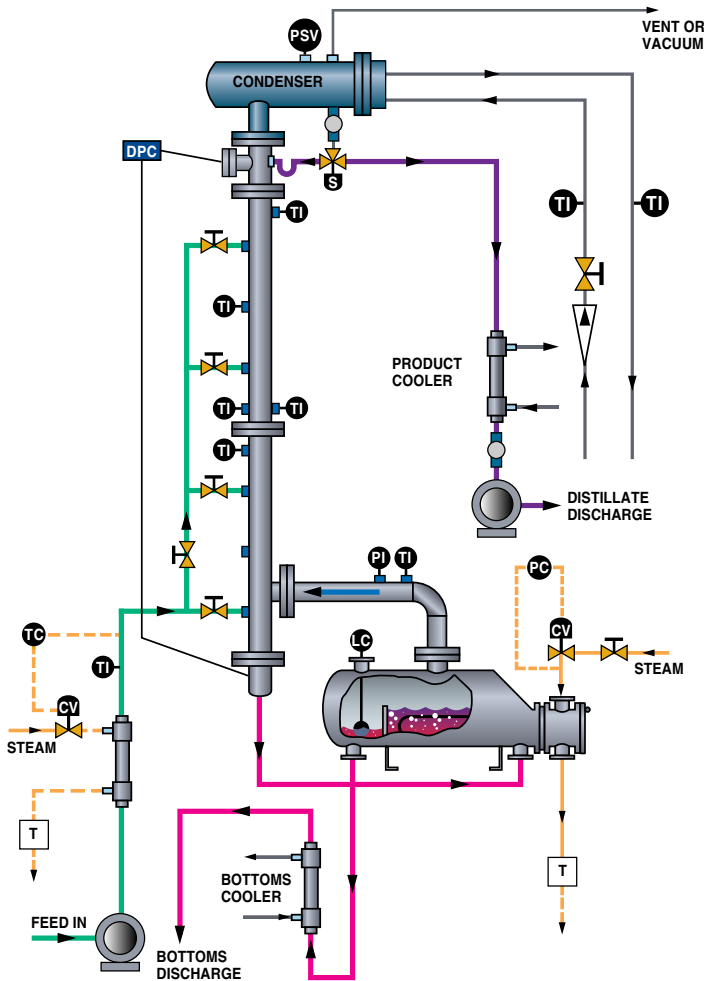
CONTINUOUS Mode

When large or continuous quantities of liquid are to be processed and/or when an extremely high level of purity is not required, continuous mode is preferable.

Standardized Component Specifications

Boiling Vessels (Batch Stills)

- All 316-L SS heavy duty construction (alternate alloys and carbon steel available).
- Sizes range from 3 gal, 9" OD to 200 gal, 36" OD.
- ASME certification stamping for full vacuum to 100 psi, 500° F is standard for sizes up to 37.4 gal. Certification optional for larger sizes.
- Low-profile horizontal configuration with 4 welded legs.
- Column connection and inspection ports with 150 lb ANSI flanges typical. Other ports are 150 lb ANSI, NPT or sanitary (alternates may be specified).
- Provided with large drain ports and valves; level, temperature and pressure sensors; liquid fill port; pressure relief valves or rupture discs and spare ports. Sight glasses and light sources are available.
- Specific surface finish and electropolishing available for high purity or pharmaceutical applications.



Typical Boiling Vessel

Typical Systems Include:

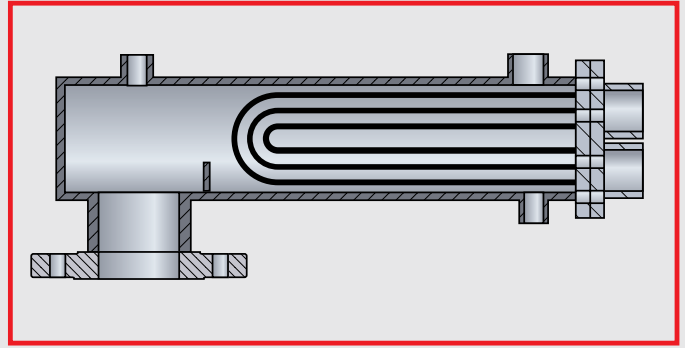
- Precision metering feed pump.
- Feed preheater section with feedback control.
- Valve manifold for selection of feed point to column.
- Column section(s), pre-loaded with packing and required internals.
- High efficiency condenser with cooling water lines, metering valve and flow meter.
- 3-way automatic reflux valve.
- Distillate product cooler and sight glass.
- Distillate gear-type discharge pump.
- Horizontal kettle-type reboiler with steam or hot oil heating controls, level switch, vapor return to column, bottoms discharge port.
- Bottoms cooler.
- Control system specified and provided by Pope or by customer.
- Skid platform and support structure for system.
- Insulation.

Boiling Vessel Heating Options

- Electric strip heaters mounted to outside of vessel belly with thermocouples and high temperature power leads. Controlled by auto-tuning PID controllers with solid state relays. Insulation included. No electrical hazard class rating.
- Jacketed vessel belly for use with hot oil recirculation system or steam. Control elements only, or complete turnkey hot oil or steam systems are available for various hazard classifications. Insulation included.

Reboilers (Continuous Systems)

- All 316-L SS heavy duty construction (alternate alloys available).
- Special high performance design with horizontal, removable U-tube bundle, bottoms weir, flash expansion space and large vapor outlet.
- Heating by hot oil recirculator or steam. Control elements only or complete turnkey hot oil or steam systems are available for various hazard classifications. Insulation mantle included.
- Ports are 150 lb ANSI, NPT or sanitary.

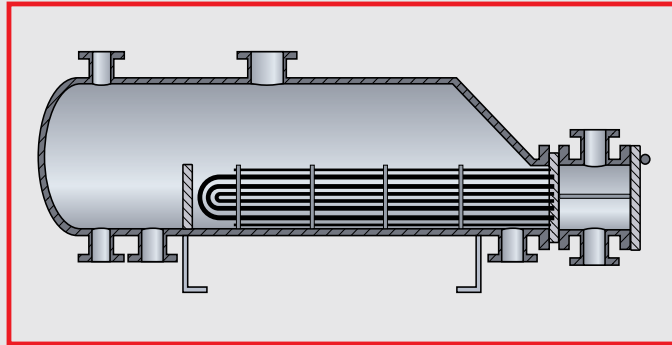
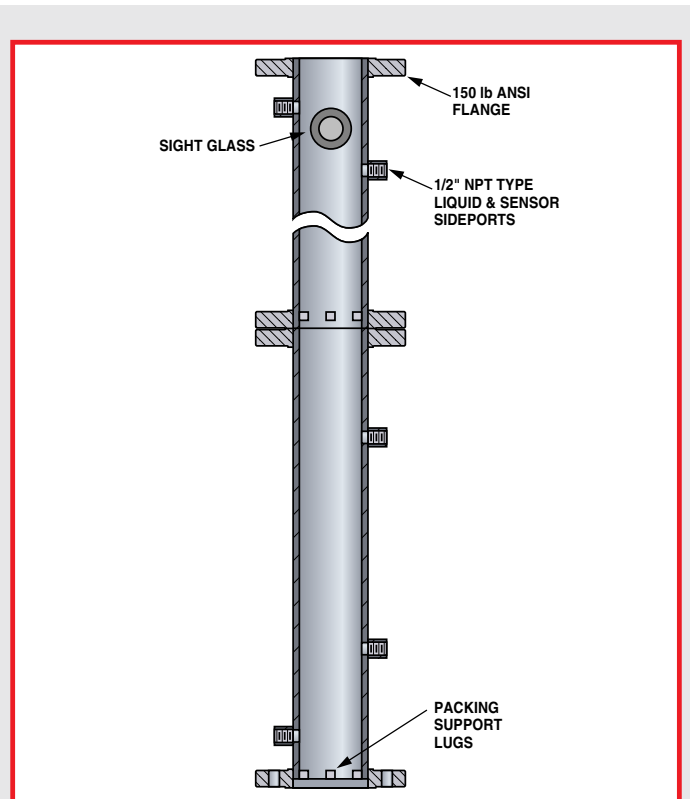


Typical Condenser

Condenser

- All 316-L SS heavy duty construction. Alternate alloys available.
- Coil bundle removable for service or cleaning.
- High efficiency, 2 or 4 pass U-tube bundle design.
- Special design for minimized subcooling and backflowing.
- Horizontal profile for low headroom.
- Spare ports for possible future modifications or instrumentation.
- Typical design ratings — 150 psi, 400° F tubes; full vacuum to 300 psi, 500° F shell. Alternate ratings and ASME certification available.
- Process connections: typically 150 lb ANSI column flange, NPT liquid and vent connections. Alternate connections including sanitary type are available.
- Water or refrigerant cooling.
- Optimized surface areas, with low pressure drop.

Typical Columns



Typical Reboiler

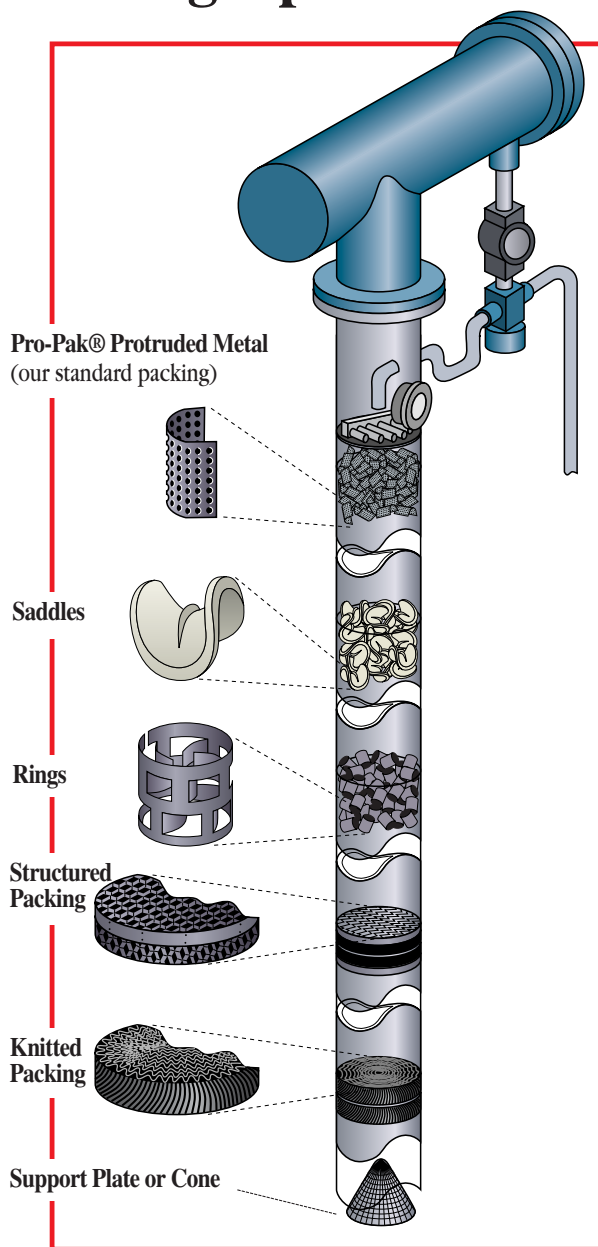
Column Sections

- All 316-L SS heavy duty construction (alternate alloys and carbon steel available).
- Diameter sizes available: 2", 3", 4", 6", 8", 10", 12".
- Sections standardized to 3' or 4' length, providing approximately 15 theoretical plates per section. Alternate lengths are also available.
- Prefabricated to accept column internals including packing supports, redistributors, hold down plates, demisters, reboiler connections, etc., as required.
- Side fittings included for liquid feed, sampling, temperature and pressure sensors. Spare ports and plugs provided for flexibility. NPT fittings are typical, alternates available.
- Main column connections typically 150 lb ANSI flanges with Teflon envelope gaskets.
- Schedule 10 or 20, typical.
- Designed for full vacuum to 100 psi, 500° F. Alternate ratings and ASME certification available.
- Welds are fine ground as standard. Mechanical and electropolishing available for high purity or pharmaceutical applications.
- Insulation included.

Column Internals

- All systems are fitted with accurately fabricated internals optimized and selected for the particular packing utilized. Internals include feed distributors, redistributors, packing hold down plates and support grids or cones.
- Particular attention is made to ensure, even liquid distribution and of low pressure drop, under all operating conditions.
- 316-L SS is standard, other materials available.

Typical Column Packing Options:



Column Packings

- A wide variety of packing types are available in the marketplace at present. Many customers have a preference for a certain type or brand name. Pope will incorporate into columns any available packing type specified by the customer.
- The standard offering is Pro-Pak® (Cannon Instrument Company), random, 316 stainless steel, 0.24" protruded packing. This is a high-efficiency packing which has been well established with over 40 years of use.
- Other random packings available include:
 - Rashig, Lessing and Pall Rings.
 - Berl and Intalox Saddles.
 - Materials include stainless steel, exotic metal alloys, ceramic and plastics.
- Non-random packings available include:
 - Woven wire mesh, spiral rolled type cylindrical sections.
 - "Structured" corrugated sheet or gauze metal cylindrical sections.
 - Bubble-cap or sieve tray plates.

As a generality, smaller diameter columns favor small size random packings such as Pro-Pak® for higher efficiency, as opposed to large size random or non-random types due to column wall effects.

Properties, Performance and Specifications of 0.24" Protruded Pro-Pak® Standard Packing

- Material of construction, 316 stainless steel (monel, other alloys available).
- 372 ft² per 1 ft³ of packing, allowing high mass transfer rates.
- 96% free space, allowing low pressure drop especially in vacuum distillation.
- Shape: half-cylinder, corners bent.
- Size: 0.24" diameter x 0.24" length, 0.003" sheet thickness, 1,024 protrusion holes per in².
- Density: 290,000 pieces and 21 lb/ft³, (316 SS).
- Packing factor: 420.
- Flood rate predicated with the following equation:

$$G=270(\rho_l)^{0.58}(\rho_g)^{0.42}$$
 where:
 - G=Mass vapor velocity, lb/ft² • hr
 - ρ_l =Density of liquid phase, lb/ft³
 - ρ_g =Density of gas phase, lb/ft³
- Efficiency of the packing increases with increasing vacuum.
- Distribution devices available for both gas and liquid to retain good operating efficiency in taller columns.

TABLE 1
Selected Comparative Column Operation with Pro-Pak® Packing

Column Diameter (Inches)	Operating Pressure	Boil Up Rate (L/hr)	Vapor Velocity (ft/sec)	HETP (Plate Height, Inches)	Pressure Drop* (Inches H ₂ O/ft)
2	atmospheric	3.7	0.35	1.77	0.12
2	atmospheric	15.5	1.46	2.10	1.70
2	(200 mm Hg)	1.1	0.31	0.79	0.12
2	(200 mm Hg)	11.2	3.20	1.33	2.77
2	(50 mm Hg)	4.6	4.70	0.92	1.35
6	atmospheric	33.0	0.36	1.76	0.14
6	atmospheric	146.0	1.59	2.57	3.36

* Pressure drop stated per foot of column height

Note: Data collected at total reflux condition

Atmospheric operations with n-heptane/methylcyclohexane test mixture

Vacuum operation with n-decane/trans-decalin test mixture

Published with permission of Cannon Instrument Company, State College, Pa.

Reflux Section

- External 3-way valve below condenser positively and accurately directs condensate to column via redistributor as reflux, or to distillate discharge as product.
- Wetted parts are stainless steel and Teflon.
- Actuated by on/off interval timer or control system.
- Required for optimizing product purity and yield.

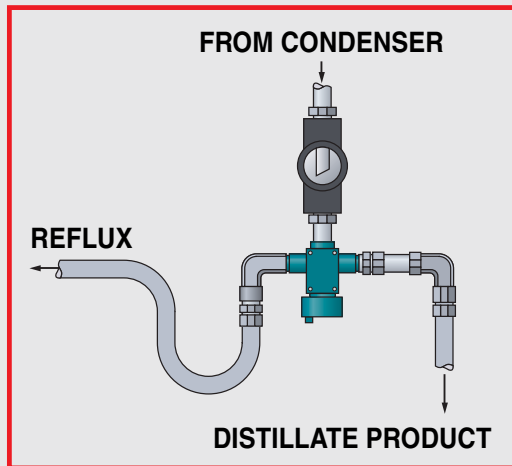


Photo shows another type of reflux device - the "swinging bucket" type, (with partial cutaway for illustration). This version utilizes an electromagnet to divert the condensed stream from reflux to distillate. No connections or valves are required, diverting is completely internal. Helps to maintain adiabatic conditions and reduces risk of leakage in critical applications. Requires a different style of condenser.

Product Coolers and Feed Preheaters

- All 316-L SS heavy duty construction. Alternate alloys available.
- Single pass tube and shell design.
- Typical design rating — full vacuum to 200 psi, 400° F, ASME certification available as an option.
- NPT or sanitary process connections.
- Water or refrigerant cooling.
- Also used in continuous systems as bottoms cooler.

Liquid Pumps

- For feeding and for distillate and bottoms discharge in continuous systems.
- For distillate discharge in batch systems (optional).
- Feed pumps are variable flow rate, heavy duty precision positive displacement type — either gear or diaphragm style. Wetted parts are stainless steel (or alternate alloys) and Teflon. Explosion proof motors are offered.
- Discharge pumps are gear type.
- Pump systems include adjustable flow controls and valve manifolds as required.
- Flow meters and sight glasses are available.

Optional Feed or Receiver Vessels

- All 316-L SS heavy duty construction. Alternate alloys and carbon steel available.
- Sized to particular application.
- ASME certification stamping for full vacuum to 100 psi, 400° F is standard for sizes up to 37.4 gals. Certification optional for larger sizes.
- Provided with ports and components as specified per particular application.
- Mechanical surface finishing and electropolishing available for high purity and pharmaceutical applications.

Mounting and Support

Systems may be supplied with a complete skid-mounted support frame or with installation on site with Pope supervision or full contracting. Systems less than 20 ft in height are generally available with prefabricated, assemble-in-place support frames. Skid and supports are carbon steel, primed and painted. Stainless steel fabrication is also available.

You may also elect to perform all mounting and installation in-house. For such cases, Pope will supply components-only systems, partial systems, or individual components.

Control Systems

Flexibility and accommodation to your particular requirements are key. Pope will supply still systems with no control elements whatsoever, or with sensors and control elements where in-house control systems are to be utilized. We can provide recommendations regarding control schemes or leave this completely to your discretion and specification.

Pope also offers systems with complete or partial control and will accommodate a wide range of requirements and budgets. Simple control loops and instrumentation for basic functions may be supplied. Alternatively, still systems may be provided with complete turnkey control and data logging consoles, with or without the utilization of PLC's or computers. For batch systems, our microprocessor-based Model 3000 Batch Distillation Controller is available which supervises distillation runs automatically from startup to shutdown. (Specifications are available upon request). Control systems can be designed to meet specified hazard classifications.

Variables Affecting System Design and Performance

TABLE 2

Examples of interaction between feed concentration, product purity, boiling point differences, reflux ratios and number of column stages (plates) required.

Initial Feed Concentration	Boiling Point Difference = 10°C		Boiling Point Difference = 15°C		Boiling Point Difference = 40°C	
	Distillate Purity = 95% Reflux Ratio	No. of plates Required	Distillate Purity = 95% Reflux Ratio	No. of plates Required	Distillate Purity = 98% Reflux Ratio	No. of plates Required
10%	47	44	28	32	2.4	13
50%	9	34	5.5	23	0.7	7
70%	6	29	3.5	15	0.4	6

Table 2 assumes a simple 2-component, "binary" application with no other contaminants. Feed concentration is the mole % of distillate in the starting material. Distillate is assumed to be the desired product in these cases. Reflux ratio is the volume directed back down the column per one unit volume collected as distillate. These examples are simplified. Many other factors besides boiling point spread affect final purity, including:

- liquid and vapor interaction between compounds
- azeotrope formations
- availability of vapor equilibration liquid vapor equilibration data
- "non-ideality" of mixture

TABLE 3

Examples of the effect of liquid density and its vapor density on the maximum reboil rate of a 2" column.

Liquid	Liquid Density lb/ft ³	Vapor Density lb/ft ³	Flooding Rate* lb/hr x ft ²	Reboil Rate lb/hr	Heat of Vaporization BTU/lb
Methanol	49.4	0.089	860	22.4	473
Acetone	49.4	0.162	1,115	29.0	224
Water	62.4	0.050	741	19.3	971

Data utilizing Pro-Pak® 0.24" protruded packing
Vapor densities at respective boiling temps., 1 atm.
*Rate per ft² column cross-sectional area

Data is often available, but in many cases it is not. Prediction is possible, but trial distillations are frequently necessary.

For instance, as shown in Table 3, when distilling acetone, the capacity is 1.5 times greater by mass than water. However, the energy required to evaporate the water would be more than three times that of the acetone.

These are just a few of the factors which must be taken into consideration during distillation system design.

TABLE 4

Examples of the effect of reflux ratios on nominal distillate production rates for different column diameters.

Column Diameter Inches	Max. Flow Gal/hr	Design Flow	Distillate Production in Gal/hr					
			Reflux Ratio 0.5	Reflux Ratio 1	Reflux Ratio 2	Reflux ratio 5	Reflux Ratio 10	Reflux Ratio 20
2	4	3.3	2.2	1.6	1.1	0.6	0.3	0.2
3	9	7.5	5.0	3.8	2.5	1.3	0.7	0.4
4	17	13.8	9.2	6.9	4.6	2.3	1.3	0.7
5	27	21.4	14.3	10.7	7.2	3.6	2.0	1.0
6	39	30.9	20.6	15.4	10.3	5.2	2.8	1.5
8	69	55.2	36.8	27.6	18.4	9.2	5.0	2.6
10	108	86.4	57.6	43.2	28.8	14.4	7.9	4.1
12	160	128.0	85.3	64.0	42.7	21.3	11.6	6.1

Based on distillation of typical light organic compounds

TABLE 5

Examples of nominal flooding capacities of various compounds with different column diameters.

Chemical Compound	Heat of Vaporization BTU/lb	Flooding Capacity in Gal/hr							
		2" Col Dia	3" Col Dia	4" Col Dia	5" Col Dia	6" Col Dia	8" Col Dia	10" Col Dia	12" Col Dia
Diethyl Ether	154	4.8	9.4	16.0	24.7	35.5	61.1	96.6	135.2
Acetone	224	4.0	7.8	13.4	20.7	29.7	51.1	80.0	113.1
Cyclohexane	154	4.6	8.9	15.3	23.6	34.0	58.4	91.5	129.3
Octane	128	5.2	10.5	17.3	26.7	38.5	66.1	103.5	146.4
Benzene	170	4.3	8.3	14.3	22.0	31.7	54.4	85.2	120.4
Ethanol	367	3.5	6.9	11.8	18.3	26.3	45.2	70.8	100.1
Butanol	254	4.1	8.0	13.7	21.0	30.4	52.2	81.7	115.5
Water	971	2.1	4.1	7.1	10.9	15.7	26.9	42.1	59.6
Methanol	473	3.1	6.1	10.3	15.9	22.9	39.4	61.7	87.2

Nominal = 80% of maximum capacity
Calculated data for 0.24" Pro-Pak®, atmospheric conditions
This information has been condensed and simplified

Pope Scientific, Inc.

Below are typical examples of Pope's Fractional Distillation Systems. All have been manufactured to Pope's rigorous quality standards and are suitable for critical high purity or sanitary applications.



Skid Mounted Continuous Purification System with 3" and 4" fractionating columns, plus absorbers, dessicator columns and control elements.



Turnkey Batch Fractional Distillation System with 12" column, 250 gallon boiling vessel, receivers, and explosion-proof PLC based control system.



Pope Hybrid Distillation System, incorporating 12" diameter fractionating column with 10 square foot wiped-film evaporator for purification of heat-sensitive compounds. System also incorporates Molecular Still and explosion-proof control system.



Complete turnkey 2-stage pressurizable **Continuous Fractional Distillation System** with 1 1/2" and 2 1/2" columns, thermosiphon reboilers, transfer pumps and receivers, explosion-proof PC-based control system.



Skid-Mounted Purification Plant, with 2" diameter fractionating column, reboiler, condenser, vaporizers, pumps, control elements, vessels and other components.

In addition to complete systems, Pope offers **individual distillation components**. Shown here, 316-SS 12" fractional column with internals, 200 gallon boiling vessel.



Pope's Autocol™ Glass Fractional Stills for batch laboratory work, small scale processing or solvent recovery. Complete automated packaged systems, 5, 12 or 22 liter sizes. (See Autocol brochure).



Batch Fractionating System with 2 gallon boiling vessel, 1" column, condenser, all stainless steel.



Solution Driven.

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