CHEMTECH SERVICES

LABORATORY DISTILLATION SYSTEMS

CHEMICAL PROCESS EQUIPMENT
PILOT PLANTS &
DISTILLATION TECHNOLOGY
Chemtech Services, Inc., designs and manufactures distillation equipment based upon proprietary technology which includes Thin Film Evaporators (TFE), Wiped Film Evaporators (WFE) and Short Path Evaporators (SPD). Chemtech’s technology roots in this field trace back over 60 years when early developments conceived by the vacuum pump manufacturer Leybold-Heraeus created continuous distillation evaporators which operated under high vacuum distillation conditions. The Leybold technology was subsequently acquired by UIC, a USA corporation, in 1989 and ultimately acquired by Chemtech.

The scope of this brochure is to describe the operating characteristics of different types of distillation equipment manufactured by Chemtech with a specific objective of defining different laboratory systems available with associated options which are available to customize a system to the customers technical requirements.

Before launching into the equipment specifics, a brief introduction associated with the applicability of thin film distillation methods to state-of-the-art chemistry is appropriate. Indicated below are some of the general field in which thin film distillation technology is currently used. Reviewing the indicated product field immediately suggests the wide applicability of thin film vacuum distillation methods. One of the most important features associated with high vacuum distillation technology relates to the reduction in the temperature at which any substance will boil.

The distillation of heat sensitive materials can be complicated; especially when the temperature at which distillation must be performed is high enough to cause thermal decomposition. Although the risk of thermal damage can be greatly reduced by distillation under vacuum, the duration of exposure to even moderate temperatures must also be considered. While, in most cases, the risk of thermal decomposition increases exponentially with temperature, it also increases linearly with the duration of thermal exposure.
Wiped Film and Short path distillation methods address each of these factors by permitting distillation at the lowest possible pressure with the shortest possible residence time. Unlike conventional "pot" distillation, the residence time of heat sensitive materials in wiped film or short path distillation is limited to the time it takes the mixture being distilled to travel under force of gravity from the top to the bottom of the heated evaporator surface. Though this depends somewhat of the viscosity of the material being distilled, the residence time of heat sensitive materials in a wiped film or short path distillation unit is generally less than 1 minute.

As the material to be distilled (or the feed product) enters the evaporator of a wiped film or short path distillation unit, it is immediately distributed as a uniform thin film on the heated interior surface, through the action of a “wiper system” which rotates within the evaporator to assure a consistent film distribution on the evaporative face of the evaporator. As gravity draws the film downward, hot oil circulating through the surrounding jacket of the evaporator transfers enough heat to the thin film to cause more volatile components of the feed mixture to evaporate. Once liberated from the surface of the thin film in this way, the resulting vapors travel to an external condenser in the case of a WFE or to the internal condenser when a SPD is used, where they are returned to the liquid state and travel downward to be recovered as distillate. Meanwhile, the remaining constituents of the film are agitated by three strands of the rotating wiper rollers which serve to maintain film thickness and uniformity while aiding the diffusion of more volatile components to the surface of the film where their concentration has just been reduced by evaporation. All components of the mixture remaining in the film as it reaches the bottom of the heated evaporator surface are recovered as residue. During the duration of the distillation, only that portion of the original feed material comprising the thin film traveling down the evaporator wall is exposed to the temperature required to effect distillation. The rest of the feed material remains in the feed vessel at a temperature which will not damage the molecules to be distilled.

Another unique feature of the “Wiped Film Evaporator” and “Short Path Evaporator” relates to their ability to achieve high Atmospheric Equivalent Boiling Point (or AEBP) temperatures through the combination of a heated jacket and the application of high vacuum conditions. Typically, the WFE or SPD can operate at temperatures as high as 350°C and absolute pressure less than 1 mm Hg (or 0.001 mm Hg in the case of a Short Path Evaporator) which conditions equate to AEBP’s of 585°C or 745°C achievable for the WFE and SPD, respectively. The Short Path Evaporator has the advantage of the close proximity of the internal condenser to the heated evaporator surface which allows the immediate transition from the vapor to liquid phase thus eliminating molecular impedance to achieving high vacuum conditions in the evaporator (and resultant high AEBP). The more conventional wiped film or falling film distillation systems utilize an external condenser, which limits the lowest pressure (vacuum) at which the evaporator can operate as the condition of molecular impedance in the evaporator are implicit in the pressure drop across the connection between the evaporator in which the concentration of vapors is high and the condenser in which the concentration of vapors is much lower.
This brochure will address primarily distillation systems based upon thin film technology, and more specifically Wiped Film and Short Path Evaporators with the objective of describing their functionality and versatility related to chemical distillations. The brochure will also describe the variety of options which can be included in any laboratory distillation system to accommodate the most difficult of separations in a cost effective manner.

Initially, it is helpful to describe the exact meaning of “Thin Film Distillation.” Thin Film Distillation is accomplished on apparatus which distributes the chemical compound to be distilled into a thin film (typically less than 1.00 mm thick) across the evaporative surface to facilitate separation (through boiling) of a specific molecular fraction for subsequent condensation. Thin film evaporator systems typically include a heating facility to initiate boiling and a condenser to re-condense the resultant vapors. Vacuum may also be applied which has the benefit of reducing the observed boiling point of the material to be distilled.

There are a number of different types of thin film evaporators commonly used in industry today including: (1) Falling Film Evaporators; (2) Packed Degassers or Flashers; (3) Wiped Film Evaporators; and (4) Short Path Evaporators. The diagram below illustrates the relationship between the most common thin film evaporators. A primary characteristic differentiating thin film evaporators is the method used to create the thin film. Falling Film Evaporators, Degassers and Flashers normally rely on non-moving parts such as plates or nozzles to distribute the product film across evaporative surfaces. Conversely, Wiped Film Evaporators rely on mechanical wiper systems, usually motor driven, to “wipe” the product into a thin film across the evaporative surface. The utility of wiped film evaporators is considered much greater than other thin film evaporators since mechanical wiping systems are capable of achieving very uniform film thicknesses even when used with highly viscous products.

The Short Path Evaporator is an even more specialized WFE, in that this evaporator utilizes an “internal condenser” positioned inside of the evaporator, and in the proximity of the evaporative surface. Locating the condenser inside of the evaporator adjacent to the evaporator wall facilitates rapid transition of the vaporized molecule from the vapor phase to the liquid phase. Phase transition is so rapid that the number of molecules present in the vapor phase (inside of the evaporator) is so small the resultant absolute pressure can be reduced to a level of 0.001 mm Hg (high vacuum). This compares with the traditional WFE/External Condenser system which typically operates at 1.00 mm Hg.

**Universe of Thin Film Evaporators**
The previous page described in general terms different types of Thin Film Evaporators. A few illustrations will facilitate understanding related to the physical appearance of these evaporators as well as demonstrating the process flow characteristics and utility requirement. Illustrated below are drawings showing a laboratory scale: (1) Falling Film Evaporator; (2) Wiped Film Evaporator; and (3) Short Path Evaporator.

The FFL Series of Laboratory TFE Systems (FFL signifies “Falling Film Laboratory) are typically configured similar to the above illustration. Generally the Falling Film Evaporators have no moving parts (like wiper systems) and rely on a dispersion plate to facilitate film formation with continuity. Note condenser is external to evaporator.

The RFL Series of Laboratory WFE Systems (RFL signifies “Rolled Film Laboratory) are typically configured similar to the above illustration. Rollers are used as an alternative to wiper blades with the added benefit of additional evaporative surface and an agitation effect which facilitates the evaporative process. Note condenser is external to evaporator.

The KDL Series of Laboratory Short Path Evaporator Systems (KDL signifies “Knudsen Distillator Laboratory) are typically configured similar to the above illustration. Similar to the RFL type evaporator, Rollers are used as an alternative to wiper blades with the added benefit of additional evaporative surface and an agitation effect which facilitates the evaporative process. Note condenser is internal to evaporator reducing molecular impedance to high vacuum.
Although Chemtech manufactures all types of distillation systems, the company is best known for its Wiped Film Evaporator based equipment. Basically, there are two popular types of wiped film evaporators utilized in the majority of the distillation systems Chemtech produces: (a) the Rolled Film Evaporator, which uses “rollers” as the wiper system used to produce a “thin film” and (b) the Short Path Evaporator, which is a subset of wiped film evaporator designed with an internal condenser to achieve high vacuum and associated high AEBP’s (atmospheric equivalent boiling points) in the operation of products. The table below details some of the features associated with Chemtech’s RFL (Rolled Film-Laboratory) and KDL (Short Path-Laboratory) Series distillation equipment.

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Wiped Film Evaporator (or WFE)</th>
<th>Short Path Evaporator (or SPD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Evaporator</td>
<td>Manufactured from Borosilicate Glass or 316 SS, this is a type of &quot;Wiped Film Evaporator&quot; (often referred to as a WFE) which utilizes rollers instead of mechanical wiper blades to achieve a uniform film thickness on the evaporator wall.</td>
<td>Manufactured from Borosilicate Glass or 316 SS, this is a specialized type of WFE referred to as a &quot;Short Path Evaporator.&quot; Like the RFL evaporator, the KDL evaporator utilizes rollers instead of mechanical wiper blades.</td>
</tr>
<tr>
<td>Evaporator Surface Area (m²)</td>
<td>0.01, 0.04, 0.06, and 0.10 or Models RFL-1, RFL-4, RFL-6 and RFL-10</td>
<td>0.01, 0.04, 0.06, and 0.10 or Models KDL-1, KDL-4, KDL-6 and KDL-10</td>
</tr>
<tr>
<td>Condenser</td>
<td>Externally Mounted, sizes range from 0.015 m² to 0.500 m²</td>
<td>Internal to evaporator, size range</td>
</tr>
<tr>
<td>Wiper System (to achieve uniform “wiped film” thickness)</td>
<td>PTFE Rollers mounted on a 316 Stainless Steel support basket to “wipe” feed material into a thin film for distillation.</td>
<td>PTFE Rollers mounted on a 316 Stainless Steel support basket to &quot;wipe&quot; feed material into a thin film for distilla-</td>
</tr>
<tr>
<td>Operating Temperature (max)</td>
<td>350 °C</td>
<td>350 °C</td>
</tr>
<tr>
<td>Operating Pressure (absolute)</td>
<td>1 mm Hg</td>
<td>0.001 mm Hg</td>
</tr>
<tr>
<td>Maximum Achievable Atmospheric</td>
<td>585 °C</td>
<td>740 °C</td>
</tr>
<tr>
<td>Nominal Processing Rate (kg/hour)</td>
<td>0.01 to 20.0 kg/hr (dependant on evaporator size)</td>
<td>0.01 to 20.0 kg/hr (dependant on evaporator size)</td>
</tr>
<tr>
<td>General Comments related to selection of WFE or Short Path Evaporator</td>
<td>The RFL Evaporator utilizes an external condenser which generally does not have size or surface area constraints, and can be sized for low boiling point products where maximum condensing surface is required. However, since the condenser is external molecular impedance limits the absolute vacuum which can be obtained and the resultant maximum operating AEBP.</td>
<td>The KDL Evaporator utilizes an internal condenser (located inside of the evaporator) which is limited in size and surface area based on the internal dimensions of the evaporator. However, since the condenser is internal molecular impedance is minimized and the absolute vacuum which can be obtained is three orders of magnitude better than an RFL evaporator which as a result allows a higher maximum operating AEBP.</td>
</tr>
</tbody>
</table>
Wiped Film (RFL) and Short Path (KDL) Distillation system designed for Laboratory distillation applications are available with a wide ranging list of features and options which can significantly affect the operating capabilities of the unit. In this brochure, a number of the basic systems configurations and potential options will be reviewed. However, our experience at Chemtech’s Short Path Distillation Division has been most RFL or KDL Systems sold in recent years have been customized to some extent based on the end user’s specific distillation requirements. Some of the basic elements and potential for customization are discussed below.

**Basic Evaporator Sizing:** The primary and most important component used in any Wiped Film or Short Path Distillation system is the actual evaporator (sometimes referred to as the distillator). As indicated previously, laboratory systems typically utilize evaporators ranging in size from 0.010 m\(^2\) to 0.100 m\(^2\). Other vessels configured in a system, such as: feed tank, receivers and cold traps, are sized based on the customer requirements. The sizing of these vessels is usually dependent on feed sample quantity requirement and duration of distillation run. For example, collection vessel sizes are as small as 0.50 liter and as large as 5.0 liters are common in laboratory units.

**Feed Vessel Specifications:** A variety of standard feed vessels are available for Laboratory Distillation systems, and non-standard vessels may be available depending on requirements. Generally the material of construction used in the feed vessel will be the same as the evaporator although there are occasional exceptions to this rule. Several operating parameters should be considered when selecting a feed vessel:

- **Capacity:** Typically 1, 2 and 5 liter vessels are available
- **Heating Requirement:** Jacketed and non-jacketed vessels available
- **Metering System:** Two basic metering systems are available: (a) Vacuum feed with a metering valve between the feed vessel and evaporator; (b) Metering pump which is typically mounted on the evaporator support plate and integrated with the feed vessel.
**Hardware Support Systems:** The “Basic System” for RFL or KDL Distillation equipment (for countertop applications) utilizes ring stands for support with the ancillary equipment (vacuum pumps and circulators) positioned strategically on the lab countertop to support the operation of the Evaporator. Many customers prefer more positive support ranging from a small stainless steel cart to larger welded frames which may support a single evaporator or multiple evaporators (staged in series) and the required ancillary equipment including circulators. Welded frames normally utilize two (2) alternative materials of construction: (1) Welded Unistrut (carbon steel) coated with two-component epoxy in a color of choice; or (2) 304 Stainless Steel Square-tube (uncoated). Both frame styles have an integrated drip-pan for spill control and special features such as exhaust hoods are available as options. Frame mounted systems also include castors and levelers for convenient movement and leveling where multiple laboratory locations may be desirable.

**Temperature Control Circulators:** Heating and Cooling Circulators are used to maintain the temperature in the critical zones associated with this system. Generally, we consider five independent zones as requiring temperature control: (1) Feed System; (2) Evaporator Heating Jacket; (3) Evaporator Residue Jacket; (4) Condensing Vessel or Coil; and (5) the Cold Trap. The ability to control the evaporating temperature and the condensing temperature suggests the circulators which service these zones are the most critical (re: zones 2 and 4 in the illustration). The necessity to use circulators for the feed and residue portions of the evaporator is a function of the viscosity of the material being distilled. Generally, when the viscosity of the materials (feed, distillate or residue) exceeds 1000 cps, circulators are recommended. Circulators range in operating characteristics and selecting the correct circulation is based upon available voltage (e.g., 240 VAC @ 60Hz), power requirements related to heating and cooling (e.g., 2.5 kW heating with 800 Watts cooling), and control interface (e.g., simple PID for local control, or PID with RS232 interface for remote control). Typically, zones 1 – 3 described above are services with “heating only circulators” while zone 4 would normally be a “heating & cooling circulator: and zone 5 a “cooling only circulator.” Most heating circulators have a maximum temperature of 300°C which typically accommodates 80% of the distillation applications, but if temperatures above 300°C are required, a high temperature circulator with a sealed oil reservoir is available, which can reach 350°C with a 6kW heating capacity. If a circulator is used to service a closed loop Cold Trap (as an alternative to a cryogenic cold trap using dry ice or liquid nitrogen) the maximum temperature requirement is typically -40°C, although duel stage chillers are available which reach temperatures as low as -90°C.

**Vacuum System:** The most typical vacuum configuration for laboratory applications utilizes a rotary vane pump as the primary source of vacuum (or backing pump) which is subsequently “boosted” (or amplified) in Short Path Systems with an oil diffusion pump to attain a 0.001 mbar dry vacuum. Alternative vacuum systems might use high capacity (CFM flow rates) rotary vane pumps or small dry pumps in special cases. In addition to the pumps used to attain vacuum, a manifold system connected to the cold trap may contains bleed valves (e.g., for N2 addition), isolation valves and a port for mounting the vacuum sensor (pirani gauge).
Above are the 5 heating zones in a common RFL-6 setup. More zones are added as the setup becomes more complex.
Product Collection Vessels: The intended functionality of the Distillation System will influence the selection of the product (distillate and residue) collection method. The simplest collection method utilizes borosilicate glass flasks which may range in size from 500 ml to 5.0 liters. The flasks are interfaced with the distillate and residue nozzles on the evaporator (or evaporator and external condenser in a conventional WFE) and would normally include an isolation valve between the evaporator (condenser) nozzles which allow the operator to empty the flask when the evaporator is under vacuum. More sophisticated distillation units might use a “Rotary Carousel Sampling System” which contains six (6) individual 150 ml sample tubes housed in a four (4) liter glass vacuum vessel. This Rotary Carousel allows the operator to obtain multiple distillate and residue product samples during the distillation process which samples can also be generated at different AEBP. The Rotary Carousel is a popular option with research organizations in their efforts to optimize process conditions through multiple sampling for subsequent analytical evaluation. The size of the distillation samples required by a customer might also dictate the necessity for optimizing the process dynamics of the Distillation System by adding vacuum rated, gear metering pumps to the distillate and residue discharge nozzles. Gear pumps are especially useful if the materials being distilled are highly viscous in nature, where positive displacement pumps will facilitate higher processing rates. The use of vacuum rated, gear pumps typically allows discharge directly to containers at atmospheric conditions. Gear pumps are a common feature in distillation systems utilizing stainless steel evaporator systems where high process rates (compared to glass evaporators) are possible through improved heat transfer. Gear pumps are available with (or without) an integral hot oil jacket for high viscosity of high melt point products which must be processed at higher discharge temperatures.
**Electrical Controls & Power Distribution:** As suggested in the preceding sections, a functional distillation system includes many elements which require electrical power to operate. Summarizing, power will be required to operate: (a) Evaporator wiper system; (b) temperature control circulators; (c) vacuum pumps; (d) product feed or discharge pumps (if applicable); and (e) instrumentation (e.g., vacuum sensors, data loggers, PLC, etc.). Simple bench top systems can be assembled on a laboratory bench with a simple power strip providing electrical utility for all the components. However, in the case of frame mounted systems which typically utilize a multitude of electrical components it is more common practice to supply an electrical panel mounted on the frame for power and instrument signal distribution to the electrical components. The size of the electrical panel is typically influenced by the complexity of the distillation system (how many electrical components), control instrumentation requirements, and customer site prerequisites (e.g., NEC code and specific customer requests). Therefore, a laboratory distillation system which includes an electrical control panel may have an enclosure which ranges in size from a 12” by 12” panel to a full size 36” by 72” electrical & instrumentation enclosure.

In addition to considering the electrical enclosure and power distribution requirements, consideration should be given to the level of instrumentation and control required for the application under consideration. In the case of benchtop systems, all switches and controllers (primarily PID’s integrated into temperature circulators) are local to the ancillary component (such as wiper system motor, PID controller on hot oil or glycol circulators). In more complex frame mounted systems instrumentation and controls are located in the electrical enclosure. Basic controls might include panel mounted power switched for ancillary components and digital displays for temperature and vacuum readings. More sophisticated instrumentation and control would include Data Logging devices or PLC controls to allow non-attended operation of the distillation system. Data Logging devices facilitate accurate record keeping which can be downloaded using a USB port and thumb drive, while PLC systems include the Data Logging function but also control the distillation based upon parameters programmed by the operator (e.g., AEBP requirement). The disadvantage of the more sophisticated Data Logging or PLC system is the higher price requirement as electronic interfaces are added to the hardware package.
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