Pope Wiped-Film Stills

Temperature Control System

Bulletin No. 4

A fundamental process attribute of Wiped Film Evaporation/Distillation is the ability to evaporate fairly high molecular weight materials without heat degradation and to strip off solvents to very low residual levels. This is accomplished by reducing system pressure, to a minimum of several microns, thereby lowering the boiling temperature of the distillate, and by creating a very thin film on the evaporator wall. The thin liquid film produced by slotted wiper blades propels the liquid across the heated surface in a few seconds, minimizing exposure to the elevated temperature. A high degree of film turbulence is also created, minimizing the temperature difference between the wall and the evaporating surface of the liquid. All of these factors combine to allow operation at the lowest possible temperature, thus preserving product stability.

In order to sustain the lowest operating temperature, it is necessary to provide uniform heat flow and effective temperature control of the heated surface.

Heat energy is transferred from one body to another by one of three fundamental mechanisms; conduction, radiation, and convection. The rate at which heat is transferred is proportional to a driving force, which is a function of the temperature difference between the heat source and its ultimate destination, and a resistance to that heat flow. For heat transfer by conduction, the resistance to heat flow is a function of the material thickness and its thermal conductivity. Most metals conduct heat very well, while ceramics, organic materials, and gases conduct heat very poorly. Obviously, the thickness of the evaporator wall and its construction material have a significant bearing on the rate of heat conduction.

Heat transfer by radiation increases by the difference of the fourth power of the absolute temperature of the emitting body and receiving body, while the resistance to flow is related to a factor called emissivity.

Emissivity is a measure of the surface's ability to absorb or reflect radiation of given wave lengths. A very highly polished metal surface reflects most of the radiant heat while a dull black body, such as an asphalt road, absorbs most of the radiant energy.

Convective heat transfer is most complex and depends upon the turbulence created in the fluid regime and the mixing of molecules of different energy levels. The turbulence created depends upon the fluid velocity, fluid viscosity, the mass flow rate and the configuration of the restraining physical barriers.

All three of these fundamental mechanisms are involved in the transfer of heat in a Wiped Film Evaporator or Molecular Still. The driving force causing evaporation is measured by the temperature difference (ΔT) between the heating element and evaporating temperature of the process fluid inside the evaporator body.

The resistance to heat flow to the process fluid is the sum of the resistances through each solid material, the convective film resistance on each side of the evaporator wall and the resistance created by the fluid film flowing across the evaporator wall. Decreasing all of these resistances to heat flow decreases the response time between the application of heat and its transmission to the evaporating fluid. It also minimizes the driving force required, the temperature difference between the heating source and the evaporative temperature of the fluid inside the system.

It is common practice to heat laboratory equipment with electrical resistance heated insulated mantles. Temperature is sensed by placing a thermocouple probe between the vessel and the heating mantle. In general, temperature control is difficult for the following reasons:

A. There is substantial resistance to the flow of heat from the heating element through the mantle because of the substantial insulation thickness on the surface of the mantle.

B. The air gap between the process vessel and the heating mantle creates further resistance to the flow of heat.

C. The capacitance (heat holding ability) of the mantle always exceeds the ability of the vessel to absorb heat by a substantial amount, causing temperature cycling and subsequent overheating with simple on/off control systems.

To allow better temperature control three heating techniques are recommended and are now available on Pope Wiped-Film Stills:

1. An electrical resistance heater imbedded in a silicone rubber mat
2. Mica insulated band resistance heaters
3. Jacketed still bodies with forced recirculation heating fluid

Each of these systems should be controlled by a controller with appropriate features, such as: proportional control, derivative response, and automatic reset. Each of these features allows very close temperature control, eliminating the usual over peaking and excessive cycling which occurs with a single on/off controller.

The conditions under which each of these heaters are to be used are described in the following discussion.

Silicone Rubber Mat Resistance Heaters

When heating temperatures of the heat source are not expected to exceed 400 degrees F. and the total heat flux is calculated to be less than 8 watts/square inch (4,913 BTU/square foot x hours), the silicone rubber mat, covered with an insulation blanket, is very practical. A typical installation is depicted in Figure 1. The mat is wrapped tightly around the still body and held in place with a Velcro seal. It makes very good contact with the body, eliminating the conventional air gap which prevails with the usual mantle type heater. For effecting better control, a heat conducting thermal cement can be placed underneath the mat, to completely eliminate the air gap which causes some resistance and interference with extremely good temperature control. The heat conducting cement has a thermal conductivity approximately twice that of borosilicate glass. The temperature sensing thermocouple is vulcanized into the mat during its manufacture, giving temperature response which will be quite close to the outside wall temperature of the evaporator body.

In those instances in which a temperature profile is required across the still body, two or more heating mats, each with its own controller, can be used. Such a temperature profile is required when evaporating a solvent from a system at a given temperature to be followed by a required increase or decrease in temperature, to alter the viscosity characteristics of the solvent-free feed material. Additionally, temperature profiles could be required when one is trying to duplicate a given reaction by providing energy of activation in the first heating area, to be followed by a different sustaining temperature down the remaining portion of the evaporator body.

One cannot arbitrarily say that the silicone rubber mat is suited for either molecular distillation or solvent stripping, since the conditions under which it is used are entirely dependent upon the heat load, and temperature of operation. Each is dependent upon the pressure of the system and the degradation temperature of the material being processed.

Refer to Table 1 for the characteristics of the silicone rubber heating mat.

Band Resistance, Mica Insulated Heaters

When the temperature of the heater is anticipated to exceed 400 degrees F., band heaters should be used. Heat fluxes of 7.5 watts/square inch to 30 watts/square inch (3,685 BTU/square foot x hours to 14,740 BTU/square foot x hours) are possible by merely changing the series-parallel wiring of multiple band heaters. With these heaters, it is possible to build in a temperature profile across the evaporator body by having individual thermocouples placed under each band heater, with each thermocouple responding to its own individual controller. Because the band heaters have a steel surface rolled to a specified diameter, the contact between the heater and the body wall is not precise. Again, to improve conduction from the heat source to the evaporator body wall, a thermal conducting cement is placed between the band heater and the evaporator wall. The sensing thermocouple for each controller is placed in the thermal conducting cement. Under these circumstances, the resistance to heat flow is minimized and the temperature control can be quite precise. A typical installation is depicted in Figure 2. (The characteristics of this type of heating system are shown in Table 1.)

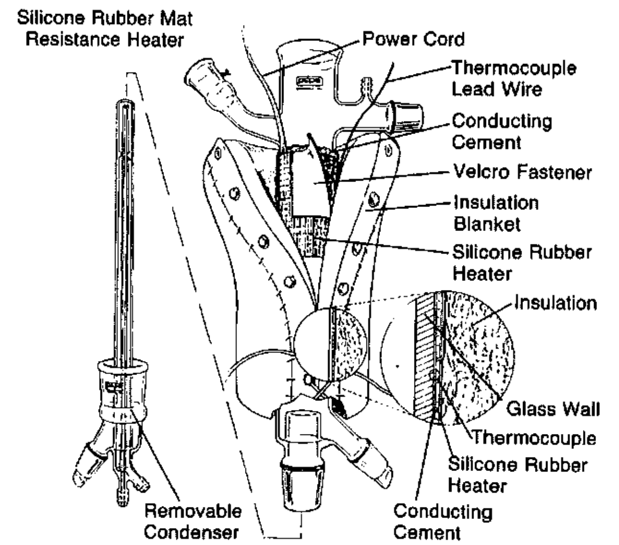

Figure 1: *Silicone rubber mat resistance heater.*

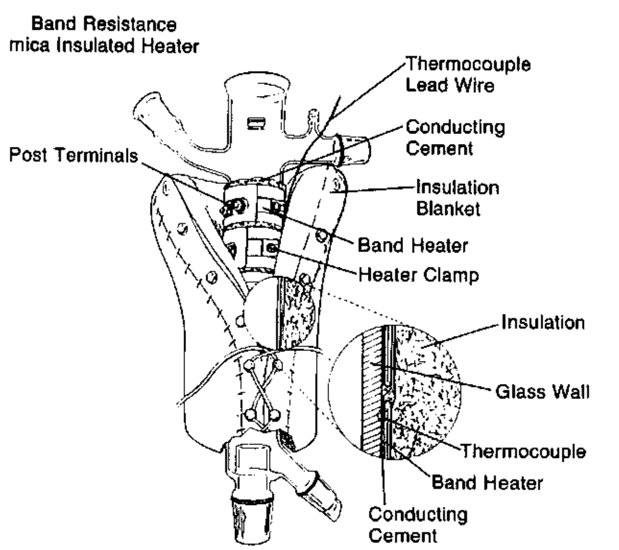

Figure 2: *Band resistance, mica insulated heater.*

Jacketed Thin Film Evaporators

While the electrical heating systems provide the greatest flexibility and most economical operation, there are instances in which jacketed vessels are desired for explosion proof operation. While it is frequently stated that the jacketed vessel provides the most uniform temperature across the entire wall, it offers little advantage over a properly designed electrical heating system with respect to temperature uniformity, and has the disadvantage of requiring isothermal operation for the entire evaporator body.

At the present time, Pope Scientific does not build ASTM pressurized jacketed vessels. Therefore, the pressure imparted to the jacket must not exceed atmospheric pressure. However, the temperature range can be up to 500 plus degrees F., with effective use of a high temperature, recirculated, heating fluid. In these instances a heating fluid reservoir holds fluid which is pumped through an external heat exchanger, which is heated either by electrical resistance heating or steam. The forced circulation fluid flows through the jacket back to the reservoir and is controlled with conventional instrumentation applied to the heat exchange system. In many cases the temperature below 212 degrees F. will suffice for solvent stripping and molecular distillation, particularly at lower pressures. In those cases hot water is used as the recirculation fluid. To minimize heat loss, and for safety of operation, the entire heating system is insulated.

In such a system the temperature sensing element for control of the heating fluid medium is placed at the discharge of the fluid flowing out of heat exchanger. (Typical characteristics of such a system are shown in Table 1.)

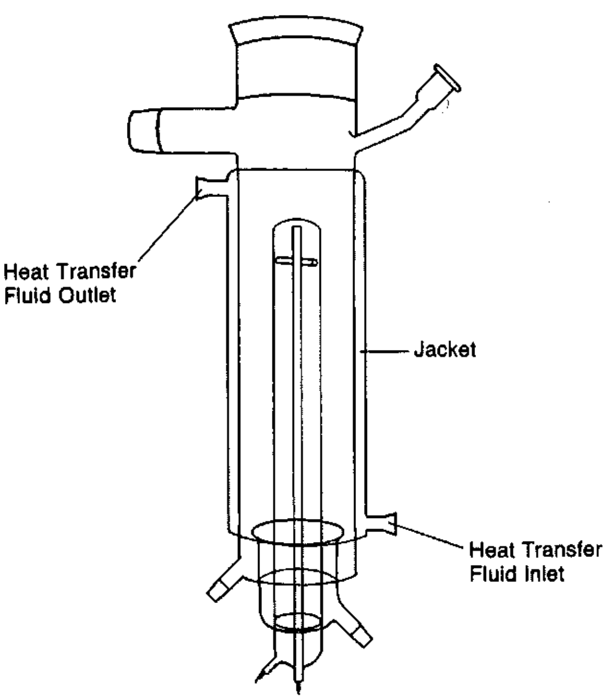

Figure 3: *Jacketed thin film evaporator body.*

Temperature Control Systems

In order to provide extremely close temperature control, it is necessary to have three key features on a controller, in addition to the simplistic on/off mode used in the least expensive controllers.

1. The controller must contain a variable proportional band setting, which is the percentage of total controller scale over which the controller turns on and off. A control system with a 500 degree F. range set at one percent proportional band, would turn on and off over a plus or minus five degree F. span. It is generally desirable to operate at the narrowest possible proportional band which allows stable control.
2. The controller should also contain a derivative response feature which anticipates the rate of rise of the temperature and, therefore, reduces the on time of the controller response if it senses the rate of temperature rise is extremely fast. It anticipates very rapid changes, causing the controller to respond to these changes, thereby minimizing temperature overshoot on system heatup. Temperature overshoot can ruin a heat sensitive product and must be eliminated in order to have an effective thin film evaporation system.
3. The controller should contain automatic reset which merely compensates for a change in total load on the system when the controller is at a given setpoint. Automatic reset allows the system to properly respond at different flow rates, but retain the same temperature setpoint.

With these three standard instrumentation features, it is possible to effect extremely tight temperature control, usually plus or minus a few degrees, even at substantially elevated temperatures. This close temperature control is possible when excessive resistances caused by air gaps between the heat source and the evaporating fluid are completely eliminated. Such elimination occurs with the proper selector of heating systems, as previously explained.

For appropriate design of a Wiped Film Evaporator application, it is necessary to determine the maximum temperature to which the process fluid can be heated. This dictates the vapor pressure of the components to be removed, the concentration of residual components, and the pressure to which the system must be reduced. The heat flux of the heating system is dictated by the quantity of material to be evaporated. All of these factors must be considered when selecting a Wiped Film Evaporator or Molecular Still.

Furthermore, it is necessary to estimate the condensing load required for the evaporated vapors and then make the judgment whether or not an internal cold finger condensing capacity is adequate or whether additional condensing capacity is required. These estimates are based on the heat transfer capabilities of the various condenser systems and the temperature of the condensing fluid available.

All of these factors are the process considerations which are used by Pope engineers to properly select a Wiped Film Evaporator for evaporation or molecular distillation.

Table I
Wiped Film Evaporator Capacities

Heater Type of	Water Distillation Rate (cc/min.) Body Diameter 2″	4″	6″
Silicone Rubber	5	17	34
Metal Heater Bands*	24	82	163
Jacketed Body (Glass)†	24	82	163
Jacketed Body (Stainless Steel)*	60	205	407

† With 400°F Heating Fluid & ΔT of 300°F

* With External Condenser

Apparatus and instrumentation for science and industry

Pope Scientific, Inc.
351 N. Dekora Woods Blvd.
P.O. Box 80018
Saukville, WI 53080 USA
(262) 268-9300 • Fax (262) 268-9400
www.popeinc.com
E-mail: sales@popeinc.com

Rev. 8/2000