9319 SHORT PATH STILL
INSTRUCTION SHEET

A short path still is a simple distillation unit which provides the shortest possible vapor path between pot and condenser in order to minimize the total heat input required for vaporization. The 9319 has a 60mm average path and practically no reflux to reheat. It is often incidental that samples are small and total holdup is required to be minimal. Drainage therfore should be maximal, with no pockets at the seals. This goal is attained in the 9319.

The use of vacuum further reduces the heat and condenser area required and is a practical necessity for distilling compounds with a normal boiling point over 175° C. Many organic compounds are thermally degraded over this temperature and particularly over 200° C.

Interferences, however, accompany distillation under reduced pressure. Dissolved gases are released with foaming, and preliminary out-gasing in a larger flask is often a prerequisite. The presence of minute amounts of lower boiling ends will produce a like result and may also induce "bumping" particularly with a pear-shaped pot that characteristically has a much larger surface/ volume ratio at the bottom and causes faster vaporization at that point.

The use of a gas bleed tube below the surface for agitation, in an effort to eliminate unequal heating and bumping, also produces a bubble structure on the surface of some liquids and inhibits vapor release at the surface. The bleed tube should be placed just above the surface in such cases to break up the bubbles and keep them in motion, but if the impinging gas stream is too cold or voluminous, it too will overcool the surface. A gas stream of about 1-2 bubbles/sec. is adequate in most cases.

Control is improved by placing a pinch clamp between the bleed tube and a 25 x 0.1mm I.D. capillary connected by a heavy wall rubber tube. (Thin walls collapse and seal) With some liquids, round bottom flasks with magnetic stirring is required to attain smooth vapor release.

Heating requires pin-point control as can be obtained with a heating bath or Instatherm; micro- burners are next to useless. A heat gun connected to a Powerstat can be operated at lower voltage: if the motor is A.C. it will overheat below half rated voltage; If D.C., lower voltages (1/4) can be used. However, heat guns are not recommended where operation is troublesome.

Contamination of product from joint lubricants can be prevented by using PTFE sleeves instead of grease. A sleeve is particularly useful on the rotating Cow receiver joint. Spray-on PTFE lubricants can be used if burnished by turning the joint when the coating is dry. Joint clips should be used for maximum safety.

When foaming is negligible and the product viscous, it is advantageous to tilt the still slightly to inhance liquid flow rate. Also, water may be partly drained from the condenser to raise condensate temperature and reduce viscosity, or air may be used as a coolant in place of water.

ACE GLASS INCORPORATED
Phone: 1-800-223-4524 Vineland, NJ 08360 **Fax: 1-800-543-6752**