

9599

SPINNING HICKMAN-HINKLE DISTILLATION COLUMN

OPERATING INSTRUCTIONS

Assembly:

Assemble the apparatus as illustrated in Figure 1. Insert PTFE Band thru male joint on bottom of column and attach pot/vial with "V" magnet of Band in pot. In the process, make sure the Band is as straight as possible. In particular, the pointed "V" section extending into the pot must be straightened so that as little vibration (wobbling) as possible occurs during rotation of the Band.

Operation:

1. Load sample (shroud sand bath or heating block with aluminum foil) and bring pot temperature rapidly to the boiling point of the lowest boiling component. Distillation will begin approximately 25-35 degrees above this boiling point. The trick to getting good results is to have the temperature rise during this range very slow. No more than 1-2 degrees/five minutes. Once collection is established, the temperature should be stabilized as much as possible (rising one degree/ten minutes). When distillation stops and temperature drops, the pot temperature is again raised to the boiling point of the next component at the 1-2 degree/five minute rate.

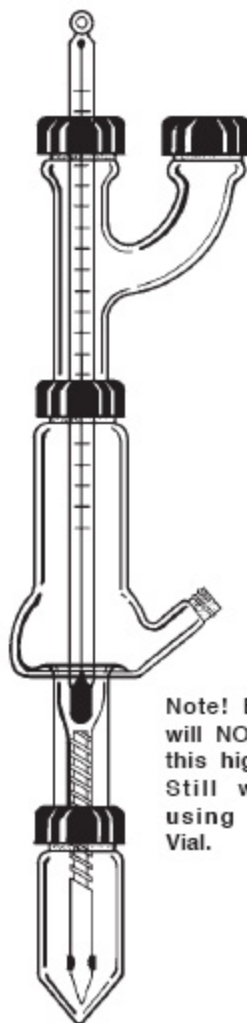
2. Accurate temperature tracking is tricky, but reliable if the thermometer is positioned just above the band as shown in Fig. 1. It should be no more than 1-2 mm above the band and as close to the bottom of the well as possible without making wall contact.

3. The temperature range for these non-reduced pressure still is 45°-111° for the 9599 Still.

4. Collection is made by removing the thermometer and inserting a Pasteur pipet or inserting a syringe through the septum in the side arm. Thermometer removal is no problem. The collection of fractions is very slow and the thermometer quickly recovers on return to the system.

IMPORTANT: Rapidly raising pot temperatures above the boiling points of the mixture components spell immediate disaster for the distillation. IT IS ABSOLUTELY NECESSARY TO MAINTAIN A VERY SLOW RISE IN POT TEMPERATURE AFTER THE VAPORS HAVE REACHED THE COLUMN, NO MORE THAN 1-2 DEGREES/FIVE MINUTES.

Figure 1



Note! Band will NOT be this high in Still when using 5mL Vial.