Operation of the Ebulliometer

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June 5, 2012

1 Introduction

An ebulliometer heats a multi-component liquid mixture to produce vapor and liquid in equilibrium. These streams can be sampled to determine composition. The ebulliometer can also measure the boiling point of liquids by measuring the temperature either isobarically or isothermally. The apparatus in the lab contains ethanol and cyclohexane. The purpose of the instrument is to provide accurate vapor-liquid equilibrium data (see Figure 1).

2 Safety

The ebulliometer contains flammable organic solvents. Use care when handling. Although temperatures do not generally exceed 76 °C for the ethanol-cyclohexane system, the glass piping becomes very hot to the touch. The heater is insulated by an additional glass layer. Always remember to turn on the condenser before the system is switched on (for at least 40 minutes to achieve a refrigerant temperature of 10 °C).

3 Startup

1. Turn on the condenser for the ebulliometer. This is done by turning on the temperature controlled refrigeration-bath located next to the ebulliometer. Getting the temperature

down to 10 °C takes at least forty minutes and the heater should not be turned on before the refrigeration bath reads 10 °C. Mike recommends turning the refrigeration on about one hour before the heater is turned on.

- 2. Make sure that there is adequate liquid in the ebulliometer system. The fluid level should be between the white mark and the elbow (see Figure 2). At the white line, the vessel contains 380 mL. Never operate the ebulliometer at a liquid level below the white line as this will expose the heating element, which could lead to damage. Do not fill the ebulliometer above the Y junction in Figure 3 or the return flow of condensed vapor will be impeded, which could result in composition errors. Fill the ebulliometer through the red cap below the condenser visible in the upper center portion of Figure 1.
- 3. Turn on the main power to the ebulliometer by switching the big, red knob to "on."
- 4. The values should be in the default position (as shown in Figures 3 and 4) to allow a recycle of the distillate to the bottom of the reboiler. The black lines on the front of the values indicate the flow path.
- 5. After the thermocouple and pressure transducer gauges display digits, turn on the heater by pressing the green button.
- 6. Wait for at least a half hour for the temperature to increase, and for the system to equilibrate, before taking measurements.

4 Taking Measurements

 To take a distillate sample, turn the top valve (vapor sample valve) so that the lines on the valve knob are aligned to permit flow from above the valve to divert to a collection vial. Collect a large enough (0.5–1 mL) sample to measure in the refractometer. Turn the valve back to its default position.

- 2. To obtain a liquid sample, first drain 15–25 mL into a waste container and then collect the sample into a vial. Be sure that the valve is not turned back to the default position between draining the recycle tube and collecting the sample. This will keep the bottoms sample uncontaminated by the returning distillate.
- 3. After obtaining the desired samples and the liquid level has readjusted, turn off the heater to allow the system to cool until the liquid in the vessel is not boiling and approaching room temperature (~30 °C). Refill with liquid by taking off the cap (be sure the o-ring remains in the cap) and pour the desired fluid into the system. Replace the cap, and turn on the heater.
- 4. During reheating, the fluid may "bump". Bumping is when vapor nucleation is hindered until a disturbance causes superheated liquid to vaporize with surprising force. This is normal for operation of the ebulliometer. However, if a sizable amount of liquid is refilled (more than 100 mL), wait at least a half hour after the vessel has stopped boiling before draining or filling the system.

5 Shutdown

- 1. Turn off the heater by pressing the red "stop" button.
- 2. Allow 30 minutes to cool. The liquid should stop boiling and be approaching room temperature.
- 3. Turn off main power to the ebulliometer and shut off the cooling fluid to the condenser.



Figure 1: Ideal Fluid Level in Ebulliometer



Figure 2: Acceptable Fluid Level in Ebulliometer



Figure 3: Gas Sample Valve



Figure 4: Liquid Sample Valve