Operating Instructions

Note: We have recently switched chemicals in the lab as follows:

- Solvent was switched from Butyl Acetate to Isopar M (a low volatility mixture of hydrocarbons).
- Solute was switched from acetone to methyl ethyl ketone (MEK)

The figures in this manual have not yet been updated and they refer to Butyl Acetate (BA) and acetone valves, lines, etc. All the items labeled as “BA” and “acetone” now correspond to Isopar M and MEK, respectively.

Preparation for Start-Up

Preparation of Isopar M/MEK Feed

1. The tank with the Isopar M/MEK feed is located on the 3rd floor and will be filled up by the lab supervisor prior to the class. The feed must be well mixed by the pump for at least 20 minutes before the experiment begins. The pump must be shut off and the valves adjusted properly before the feed is introduced to the 1st floor.

Notes:
- The feed should be well mixed before the experiment. Otherwise, you may have large fluctuations in the composition of the feed which would make it difficult to reach a steady-state.
- The MEK composition in the feed and product streams should be sufficiently high to be measured by titration.

2. To avoid flooding the columns during start-up, check that the main Isopar M and water feed valves on the 1st floor are closed. (Figure 4-1)

3. Go up to the 3rd floor, check the level in the Isopar M/MEK feed tank to make sure that there is enough feed for the lab period.
Handling Waste Water/MEK Mixture

The waste water/MEK mixture should be collected in 55-gallon drums located outside on the containment pad (Figure 4-2).

**WARNING:** Do not jump off the loading dock to access waste collection drums – use the stairs.

**Before the experiments**

1. Remove the drum wrench, the red float assembly, hazardous waste labels, and a pen from the center drawer on the right side of the desk in front of the titration area and take them outside.
2. At least two students should lift the plastic drum cover straight up until it clears the drum tops and then move it to a position on the ground near the drums.
3. Check the drum usage log and choose a (partially) empty drum. Do not start a new waste drum if there are drums that are only partially full – fill up those drums first!
4. After choosing a drum, use the drum wrench to remove the large and the small plugs from the drum.

**WARNING:** Unscrew the plugs slowly to bleed the pressure from the drum. Keep your face away from the plugs and wear eye protection.

5. Check the level of liquid in the drum by the measuring stick (located near the fume hood). Each tick mark on the stick corresponds to 1 gallon in the standard 55 gallon drum. Record the level in the log.
6. Attach the grounding clip (located in the center of the platform) to the drum to be used.
7. Place the metal hose into the large plug hole and open the waste valve (V15) by the building.
8. Screw the red float level indicator into the small plug hole on the drum, see Figure 4-3.

Figure 4-2. Waste drums
**During the Experiment**

1. Monitor the red float which begins to rise when the drum is nearly full. Note that this happens suddenly.

   Do not let the level rise above two inches from the top of the drum.

   **IMPORTANT:** One on the group members should watch the drums constantly to avoid spills.

   If a drum overflows the group must contact the lab supervisor and then clean up the spill with absorbent. This procedure takes about 1.5 hours and must be done before anyone can leave the lab.

![Figure 4-3. Waste drum setup.](image)

2. When a drum is full
   a) Date the yellow hazardous waste label attached to the drum.
   b) Open another drum and move the waste hose and indicator into it. **Be careful when transferring the hose to avoid spills!**
   c) Fill out the yellow hazardous waste label for the new drum: specify the chemicals and their approximate concentrations and attach the label to the drum. Do not date the label until the drum is full.

**After the Experiment**

1. Measure the liquid level in the waste drum and record it in the log.
2. Remove the hose, cap all plug holes with plugs, and close the waste valve (V15).
3. Place the plastic cover on the drums.
Start Up

Open the feed lines (3rd floor)
1. Make sure the mixing pump for the organic phase is turned off. Close the valve V2 and open the feed valves V3 and V4.
2. Check the valves to confirm that they are in correct positions to enable the flow of the feed from the bottom of the tank to the line passing down through the floor.

Prepare a Column (1st floor)
1. Select a column for the run. Check that the valves to and from the other columns are closed.
2. Set up the column by opening the outlet valves for raffinate (V10, V11) and the outlet valve for extract (V13) first, then open the inlet valves for water (V5, V6), see Figure 4-1 and Figure 4-4.
   Set up the column by opening the appropriate valves to direct flows to the selected column
   Note: The drain valve V14 (see Figure 4-6) for water/MEK exit stream should be fully closed before introducing the water feed into the column.
3. Introduce water by rotameter (Rw) and fill the column with water as the continuous phase. To do this, open the water shut off valve (Figure 4-1) and the needle valve on the water rotameter (Figure 4-4). Water will flow into the column from the top (Figure 4-5).
4. When the water level is about to reach the top edge of the rings, decrease the water flow rate to the desired value, and open the water/MEK drain valve (V14) and adjust it to maintain a constant water level in the column. Stabilize the water level between the rings and the raffinate exit pipe near the top. Make sure that water does not flow into the outlet pipe for raffinate at the top of the column. If this happens, we will have to separate water from the organic phase in the raffinate tank!
   Note: Adjustments to the drain valve (V14) often need to be very small, ¼ turn or less. One of the group members should be responsible for observing and controlling the water level with this valve at all times during the experiment.
5. When the level is stable, the Isopar M/MEK mixture can be introduced to the column using valves V7, V8, see Figure 4-1 and Figure 4-4.
   Note: The outlet valve (V11) for Isopar M on top of the raffinate tank must be open to avoid overflow and spill on the floor.
6. Introduce Isopar M into the column:
   a) Open the shut-off valve (Figure 4-1) for Isopar M.
   b) Control the flow rate using the rotameter RI.
   Note 1: Start with a low flow rate to learn the interface control and then increase the flow rate slowly to the desired value.
   Note 2: The objective is for the water to absorb MEK and be drained from the bottom of the column and the Isopar M collected for reuse by flowing up into the small collection tube at the top.
   Note 3: An interface between water and the organic phase will form and must be maintained at a proper position. Do not change the water flow rate. Adjustment of the drain valve (V14) should be the ONLY way to control the interface level.
If the interface level increases and you cannot control it with the drain valve, shut off the water inlet valve (V5) to drop the interface level.

Figure 4-4. Feed valve on the 1st floor.

Figure 4-5. Column setup
During an Experimental Run

1. Adjust the flow rates of water and Isopar M using the rotameters.
   
   Recommended flow rates are
   
   - For Isopar M: 5 to 15 l/h
   - For water: 15 to 25 l/h

2. Control the interface level by adjusting the drain valve (V14) to maintain the level above the top of the rings and also below the raffinate exit pipe.
   
   Note: No waste is allowed to exit with Isopar M due to the recycling of Isopar M. Interface level must be watched carefully and constantly.

3. Samples of the inlet Isopar M (feed) and outlet Isopar M (raffinate) should be taken by opening valves V9 and V12, respectively (see Figure 4-7), every 10-15 minutes until the system reaches a steady-state.

4. Determine MEK concentration in each sample by performing titration in the fume hood as described in the Titration handout.

5. Once the titration is completed, perform an MEK mass balance to determine if the column is at a steady state. You can assume that the system has reached a steady state if the flow rate of MEK entering the column is the same as the flow leaving, to within a tolerance of 5%.
Changing Columns

1. Shut off Isopar M feed by closing the rotameter R$_1$ and the inlet valve V7.
2. Close the drain valve (V14) to fill the column with water to push Isopar M at the top of the column into the raffinate tank on the 1$^{st}$ floor.
3. When the organic phase/water interface is about to reach the raffinate exit pipe, shut off the water inlet valve (V5) and close the water rotameter (R$_w$) immediately. It is better to lose some Isopar M than contaminate it with water.
4. Close the column’s raffinate exit stream valve and the Isopar M drain valve.
5. Open the drain valve V14 and drain the water/MEK mixture into the waste drum outside.
7. Close the drain valve V14.
8. Prepare another column (instructions for column preparation are given above)

Shutting Down

1. Shut down the column by following steps 1-8 of instructions for changing columns.
3. Close the Isopar M inlet valves (V3, V4) on the 3$^{rd}$ floor
4. Close waste valve (V15) outside, remove the hose, cap all plug holes with correct plugs, and attach yellow labels to the drums used.

Cleaning Up

1. Empty beakers containing samples and hydroxylamine hydrochloride solution into the
appropriate waste container to the left of the fume hood and close the container.

2. Used sample vials should be thrown into the appropriate waste container. They do not need to be emptied or have labels removed.

3. Fill out and apply yellow labels to the waste containers.
   **Note:** A yellow label attached to the waste container with vials should read “used sample vials containing Water, Isopar M, and Methyl Ethyl Ketone”.

4. Throw used Kimwipes, pipette tips, and gloves into the container for hazardous solid waste.

5. Turn off the pH meter.

6. Turn off the light to the fume hood and close the hood window.

Figure 4-8. Waste Labels. Fill in Team and Date when the container is ready to be picked up for disposal.