

Simple Distillation: Purification and Reuse of Acetone

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Chemical Concepts / Green Lessons

Distillation, solvent recycling, boiling point determination

Estimated Lab Time

1.5 – 2 hours

Scenario

The following letter was sent to *Lecher Consulting Enterprises* from Mary Standards.



Great American Paint Company
123 U.S.A. Parkway
Freedom, IN 47240

Dear Dr. Lecher,

My name is Mary Standards and I am the Vice President of Operations at the Great American Paint Company. I am looking for technical assistance with a proposal which may be of great value to our company.

At the Great American Paint Company, our focus is on making high quality paints at reasonable prices. For the last several years, we have been using acetone to clean the inside of our processing equipment between batches of different colored paint. After the acetone is used, it is contaminated with paint and must be collected and drummed. For the last several years we have been using AAA Disposal Company to pick up our acetone waste and dispose of it in accordance with state and federal regulations. The cost of the disposal service has risen dramatically over the past five years. However, we have been unable to raise the price of our paint products to offset these increased production costs due to stiff competition from foreign paint products that do not face the same environmental regulations that we face here in the United States.

It was recently brought to my attention that it may be possible to devise a procedure to recycle paint-contaminated acetone. I am asking you to propose a method of recycling the acetone, to demonstrate the method on a small scale, and then to demonstrate that the recycled acetone will be of sufficient quality to be reused.

Sincerely,
Mary Standards
Mary Standards
Vice President of Operations

Lecher Consulting Enterprises is a top notch chemical consulting firm which specializes in environmentally benign chemical techniques and procedures. You have recently been hired by *Lecher Consulting Enterprises* and have been assigned to work on the problem outlined in the attached letter.

Background

The process of vaporizing a liquid mixture in one vessel and condensing the vapors into another vessel is called distillation. The liquid being distilled is heated in a flask, which is sometimes called a *distillation flask* or *distillation pot*. The vapors are condensed on a cool surface, usually a water-cooled condenser. The resulting liquid is called the *distillate* and is collected in a receiving flask.

A vaporization-condensation cycle is when a substance vaporizes and condenses one time. A *simple* distillation involves only one vaporization-condensation cycle. Purification occurs as the volatile liquid vaporizes and leaves behind the nonvolatile (or less volatile) impurity. Purification by simple distillation is appropriate for liquids that contain either nonvolatile impurities, or very high boiling point impurities. When a liquid contains an impurity with a boiling point close to its own ($\sim 25^{\circ}\text{C}$), then fractional distillation, which involves multiple vaporization-condensation cycles, would be required to separate the two substances.

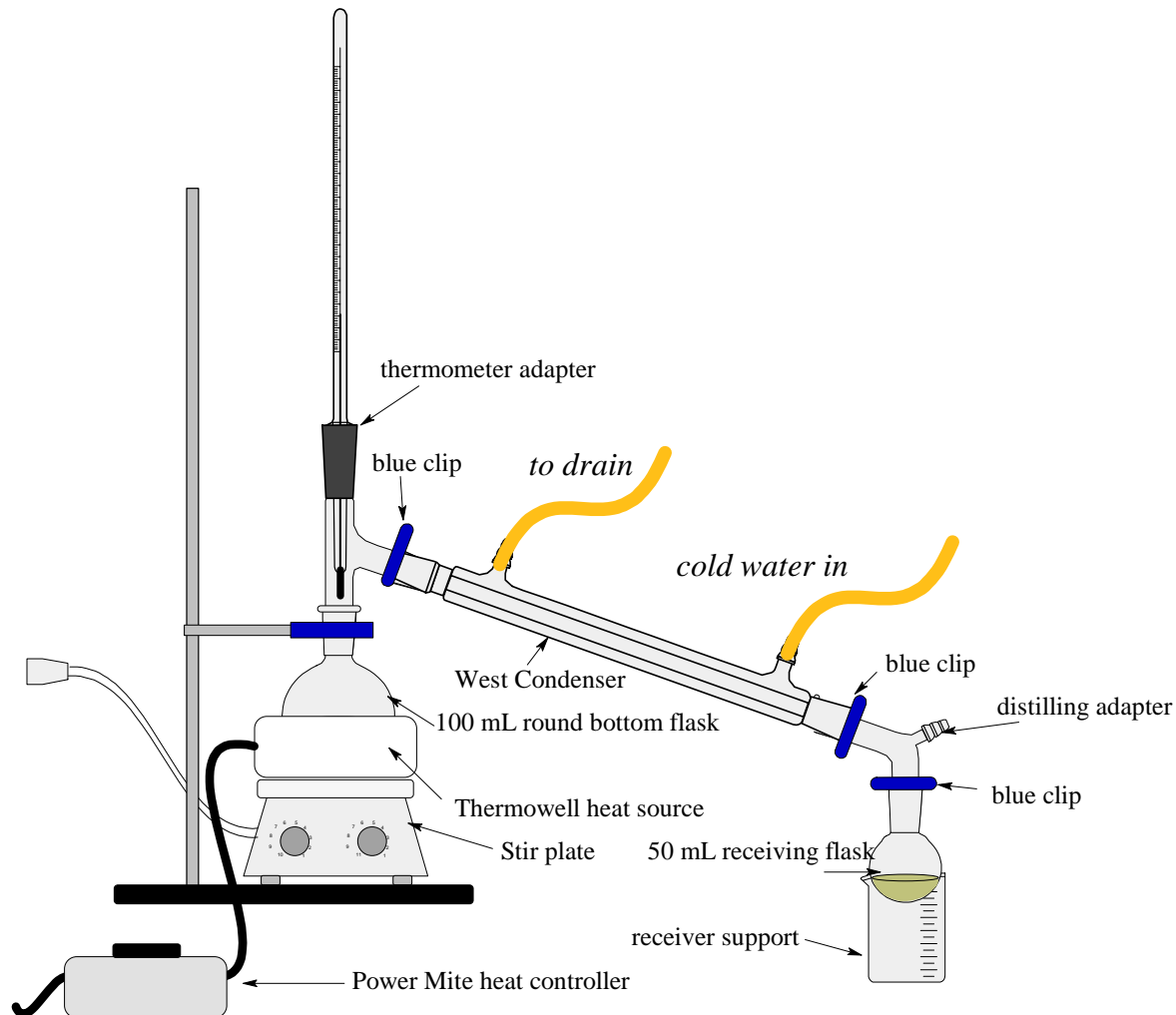


Figure 1. Distillation Apparatus

Safety Precautions

Acetone: Acetone is volatile and flammable; avoid open flames.

Experimental Procedure

Distillation of Acetone

- Assemble the apparatus as depicted in Figure 1 as follows:
 - Set up ring stand and stir plate.
 - Make sure that the Thermowell is plugged into the variable heat controller. **ALWAYS** use a variable heat controller with your Thermowell. **Never plug the Thermowell heater directly into an electrical socket!**
 - Clamp a 100 mL round bottom flask (always check glassware for cracks) into the bowl of the Thermowell. The flask should be immobile.
 - Add approximately 40 mL of contaminated acetone via a funnel.
 - Add a stir bar.
 - Attach a three way adapter.
 - Attach a thermometer adapter.
 - Carefully insert thermometer so that the bulb is slightly below the side arm of the three way adapter.
 - Using a blue clip, attach the West condenser so the jacket inlet / outlets are pointed up.
 - Attach the distilling adapter, securing it the West condenser with a blue clip.
 - Attach a 50 mL round bottom flask to the distilling adapter with a blue clip. If the 50 mL round bottom flask does not rest on the bench top, use an appropriate size of beaker to support the receiving flask.
 - Attach water hoses as depicted. Note that the condenser water should flow *in* the lower end of the condenser and *out* the upper end.
 - Turn on the condenser water supply to provide a gentle but steady stream of cooling water.
 - Have your apparatus checked by the instructor before beginning to heat.
- Begin stirring and turn on the heat source to achieve a gentle boil. Start the stirrer and turn on the heat source (~30% power). Adjust heat source so that the liquid boils gently and the reflux ring of condensing vapors rises slowly into the still head. Shortly after the reflux ring reaches the thermometer bulb, the temperature reading should rise rapidly and vapors should begin passing through the sidearm into the condenser, coalescing into droplets that run into the receiving flask. As the first few droplets come over, the thermometer reading should rise to an equilibrium value and stabilize at that value. At this time, the entire thermometer bulb should be bathed in condensing liquid, which drips off the end of the bulb into the pot.
- Record the temperature at which the thermometer reading stabilizes; if it is lower than expected, recheck the thermometer placement.
- If the initial thermometer reading is within the expected boiling range, **skip** to the next step. If the initial thermometer reading is below the expected boiling range, *then continue the distillation until the lower end of the range is reached, collecting the forerun in the receiver, and then replace the receiver by another one. Try to make the switch quickly enough so that no distillate is lost.*
- Distill the liquid at a rate of about 1 to 3 drops per second, monitoring the temperature frequently throughout the distillation. Distil until one of the following occurs:
 - the receiving flask is 75% full,
 - or until about 5 mL of volume remains in the boiling flask,
 - or the upper end of the expected boiling range is reached.

6. Turn off the heat source. Do not distil the boiling flask completely dry! Remove the heat source if necessary. Heating a dry flask will cause tar formation (or even an explosion!).
7. Transfer the remaining pot residue to the container in the fume hood labeled POT RESIDUE.
8. Clean the distillation flask with soap and water. The remaining glassware will *already* be clean from the distillation of the acetone.

Verification of Purity

9. Verify the purity of your acetone by utilizing two separate techniques which you deem to be appropriate. Review the course Operating Procedures for student procedures as well as for technique principles and applications

Disposal and Recycling

10. When you are satisfied with your purity data, place your purified acetone in the appropriate container in the fume hood labeled PURIFIED ACETONE. The acetone will be reused by the instructor.

Assignment

In addition to completing your laboratory notebook, type a business-style letter as a representative of *Lecher Consulting Enterprises* to Mary Standards of the Great American Paint Company. In your letter, address whether or not it is possible to recycle acetone. If so, propose your method of purification. Describe to her how your lab apparatus functions. You should specifically address how purification occurs (including what happens to the impurities). Additionally, you should address how the purity of the acetone was verified.

Due: _____