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Nicholas A Weires
University of California, Los Angeles


Aubrey Montebello
Boise State University


O. M. Mcdougal
Boise State University

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LABORATORY EXPERIMENT

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# Recycling of Waste Acetone by Fractional Distillation

Nicholas A. Weires,[†] Aubrey Johnston,[†] Don L. Warner,[†] Michael M. McCormick,[‡] Karen Hammond,[†] and Owen M. McDougal*[,†]

[†]Department of Chemistry and Biochemistry, Boise State University, Boise, Idaho 83725-1520, United States

[‡]U.S. Department of Homeland Security, Customs and Border Protection, 610 Canal Street, Suite 1100, Chicago, Illinois 60607, United States

*Supporting Information*

**ABSTRACT:** Distillation is a ubiquitous technique in the undergraduate organic chemistry curriculum; the technique dates back to ca. 3500 B.C.E. With the emergence of green chemistry in the 1990s, the importance of emphasizing responsible waste management practices for future scientists is paramount. Combining the practice of distillation with the message that waste generation should be minimized conveys green concepts from the beginning of the student's experience in the lab. In this experiment, acetone waste collected from the cleaning of student glassware is purified by fractional distillation. The purity of the resulting distillate is determined by refractive index and density calculation. The distilled acetone is of sufficient purity (~88%) that students can reuse it to wash glassware, collect the waste, and add it to a communal still that is operated by the instructor or support personnel. Students learn how to set up and perform a fractional distillation experiment, learn how to test the distillate for purity by refractive index and density, and are exposed to the value of recycling materials for reuse. The communal distillation apparatus provides an ongoing source of purified acetone for students to use throughout the remainder of the term.

**KEYWORDS:** First-Year Undergraduate/General, Environmental Chemistry, Laboratory Instruction, Organic Chemistry, Hands-On Learning/Manipulatives, Green Chemistry

Acetone is an abundant and useful chemical in the laboratory because of its role as a nonhalogenated organic solvent that is water miscible. Acetone is relatively inexpensive, reasonably benign, and is often the solvent of choice for cleaning dirty glassware in the undergraduate chemistry laboratory. For perspective, in 2009, ~5.1 million metric tons of acetone was produced worldwide for the principle purpose of serving as an organic solvent.[1] Because of its broad range of uses, acetone constitutes an appreciable quantity of the waste generated in the organic chemistry laboratory. Because acetone is water-soluble and it is often used for cleaning glassware, the waste is often illegally disposed of down the sink drain. Acetone waste is considered hazardous and is required by the U.S. Occupational Safety and Health Administration (OSHA) to be sent to an approved waste or incinerator facility.[2] Greater emphasis on the harmful effects of introducing organic solvents into water resources has led to an increase in the volume of acetone collected in waste bottles for disposal at the end of each academic term. In the modern era of environmentally conscious chemistry, there is a growing need to educate students in responsible waste management and the principles of green chemistry.[3] Thus, an exercise incorporating a conventional laboratory technique with the importance of environmentally friendly practices was developed.

In this exercise, acetone waste collected during previous academic terms is used to introduce the technique of fractional distillation and the three R's of green chemistry: reduce, reuse, and recycle. Students are provided 35 mL of waste acetone to purify by fractional distillation. The distillate is compared to reagent grade acetone by refractive index and calculation of density for purity. In general, students obtain acetone that is between 85% and 90% purity after one round of fractional distillation. This experiment is appropriate as the first exercise of the term in both the two-semester and the one-semester organic chemistry laboratory curriculum. It provides an opportunity to introduce environmentally friendly lab practices early in the students' laboratory experience. Instructors are encouraged to use this experiment to make students aware of how much material they use, the hazards associated with the chemicals they are exposed to in the lab, and responsible disposal procedures. This lab represents an expansion of that proposed at the University of Oregon, wherein simple distillation and student-proposed methods of analysis are utilized to assess product purity of distilled acetone.[4]

One of the most constructive aspects of this experiment is the instructor-maintained communal still that resides in the fume hood. The reflux flask is 1 L in volume, large enough for the distillation apparatus to purify waste acetone generated in the laboratory for use by multiple laboratory sections. Although having students purify their own small sample of waste acetone is valuable because it teaches laboratory technique, the real value of this exercise is to introduce the importance of recycling; an idea that will be reinforced by the instructor's continued use of the large-scale "recycling still" throughout the term. The instructor provides purified wash-grade acetone to the students, and the students provide waste acetone to feed the communal still. The students deposit their waste in the waste container in the fume hood, the instructor reloads the still prior to the next lab period, and there is freshly purified wash-grade acetone available for the next section of students. This cycle of acetone use and reuse

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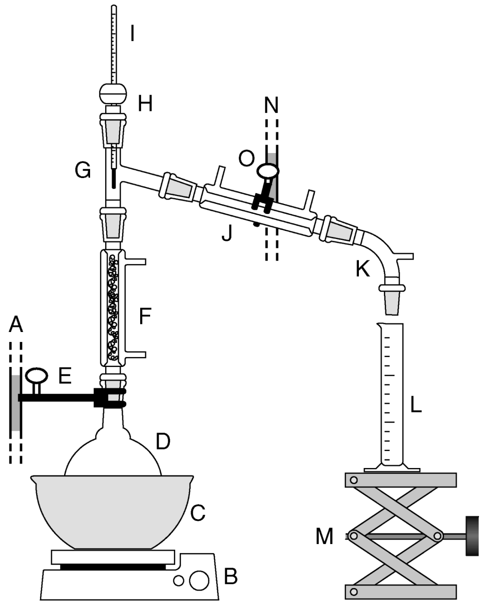


**Figure 1.** Macroscale fractional distillation apparatus: (**A**) ring stand, (**B**) stirrer—hot plate, (**C**) hot water bath, (**D**) still pot (100 mL round-bottom flask), (**E**) buret clamp, (**F**) fractionating column (a condenser lightly packed with steel sponge), (**G**) distillation head, (**I**) thermometer, (**H**) thermometer adapter, (**J**) condenser attached to (**N**) a second ring stand via (**O**) a second clamp, (**K**) vacuum adapter, (**L**) 50 mL graduated cylinder (acetone receiving vessel), and (**M**) a lab jack to hold the receiving vessels in place.

greatly reduces the environmental impact of the organic chemistry lab.

## JUSTIFICATION

This lab is intended to introduce the technique of macroscale distillation with emphasis on the green chemistry mantra initiated by the Environmental Protection Agency: reduce, reuse, and recycle.[5] Students learn how to responsibly manage waste and minimize its production. Successful completion of this lab provides students with the skill-set required to perform a fractional distillation, gain awareness for their use of solvents, assess their exposure to those solvents, and learn how to properly dispose of organic chemicals.

## MATERIALS

To perform this experiment, an organic chemistry lab must have a balance and a refractometer so that students can calculate density and obtain a refractive index of their distillate and pure acetone. In addition, a lab should have enough glassware for each student to perform the experiment. The required supplies for each student are listed in the Supporting Information and a diagram of the distillation apparatus as it is to be constructed is shown in Figure 1.

## EXPERIMENT

The students begin the experiment by constructing the macroscale fractional distillation apparatus as presented in Figure 1. A detailed discussion of distillation apparatus assembly is provided in the Supporting Information. Students begin with ~35 mL of waste acetone. The refractive index of the waste sample is taken

**Table 1. Compiling Density Information for Waste and Distilled Acetone Samples**

| Quantity | Value |
|---|---|
| Mass of Empty Still Pot/g | |
| Mass of Still Pot + 35 mL Waste Acetone/g | |
| Mass of 35 mL of Waste Acetone/g | |
| Mass of Empty Receiving Vessel/g | |
| Mass of Receiving Vessel + Distillate/g | |
| Mass of Distillate/g | |
| Volume of Distillate (Read From Graduated Cylinder)/mL | |

**Table 2. Comparison of Acetone Literature Values to Those of the Distillate**

| | Acetone[a] | Distillate | Waste Acetone |
|---|---|---|---|
| Boiling Point/°C | 56.5 | Range: | N/A |
| Refractive Index | 1.3600 | | |
| Density/ (g/mL) | 0.785 | | |
| Mass % Acetone | N/A | N/A | |

[a] Literature data from ref 6.

and the density of the waste sample is calculated from the sample mass and volume. The student instructions describe the use of a refractometer and provide information related to the meaning of the refractive index value.

Students transfer the acetone into the still pot, a 100 mL round-bottom flask, and heat is applied via a water bath—hot plate heat source. Students collect the non-acetone condensate that distills off below 50 °C. A clean preweighed graduated cylinder is then used to collect the acetone fraction between 50 and 58 °C. The graduated cylinder is utilized in this exercise because it allows instantaneous volume reading of distillate without glassware transfer; a value that students will require to calculate the density of the distillate. It is recommended that the mouth of the graduated cylinder be placed above the level of the vacuum adapter (K) to minimize fumes. An additional recommendation is to wrap the vacuum adapter and mouth of the graduated cylinder with plastic wrap to ensure fume containment. The still pot should be monitored to ensure a minimum volume of 5 mL is retained. In the event the quantity of impure acetone in the still pot becomes questionable, the heat should be immediately removed to prevent heating to dryness.

After collecting the acetone fraction, students calculate the density and obtain the refractive index of the distillate. Students also compare the mass of the acetone distillate to the original mass of the waste acetone sample to calculate a rough mass percent composition of acetone in the waste sample. At the conclusion of the experiment, the distilled acetone is used by the student to refill their individual acetone wash bottle. Once the contents of the wash bottle are depleted during the course of the term, the student can obtain more recycled acetone from the communal acetone wash still maintained by the instructor in the hood. All mass, density, refractive index, and mass percent composition values obtained by the students during the experiment are recorded in Tables 1 and 2 (included here as they appear in the procedures in the Supporting Information). The refractive index listed in Table 2 is temperature and pressure dependent. Students are provided the background information in

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the student instructions to correct their recorded refractive index for the temperature and pressure in the lab at the time of the experiment.

## HAZARDS

Acetone is extremely flammable as both liquid and vapor and may cause a flash fire upon heating. The use of a water bath and hot plate minimizes this risk as compared to direct heating under open flame; a practice that is not recommended. Acetone vapor is harmful to breathe and causes irritation to the respiratory tract. Both the liquid and the vapor cause irritation to the skin and eyes and can affect the central nervous system, so care should be taken to minimize physical contact and avoid breathing the vapors. Acetone should be handled with proper personal protection including safety goggles and gloves. The hot fractional distillation apparatus is capable of inflicting burns. The heat source should be removed if only 5 mL of the original mixture remains in the still pot. Overheating the residue left after a distillation may lead to an undesirable flash fire.

## DISCUSSION

This is the first experiment in the organic laboratory. Students perform the experiment in a standard 4-h laboratory period. The approximate times for each step of the experiment are (i) recording of a refractive index, 2−12 min, avg. 5 min; (ii) distillation apparatus set-up time, 10−45 min, avg. 23 min; (iii) distillation time to first drop of distillate, 7−72 min, avg. 31 min; and (iv) total time of distillate collection from first drop until completion, 33−136 min, avg. 106 min. These values are based on a survey of organic chemistry laboratory consisting of 24 students. The average time from start to completion of the experiment was 170 min or 2 h and 50 min. Students compare the values they determine in the lab to those provided in Table 2. Typical densities for waste acetone prior to distillation range from 0.6450 to 0.9035 g/mL with an average of 0.7966 g/mL based on two laboratories ($N$ = 36). The density values for student distilled acetone ranged from 0.7390 to 0.8530 g/mL with an average of 0.7880 g/mL. Average values for waste acetone to distilled acetone refractive indices changed from 1.3643 to 1.3618, respectively. The distillation reflux temperatures for acetone ranged from 49 to 58 °C with a typical temperature change during student collection of approximately 3 °C. The percent composition of acetone in the waste was reported by students to range from 80.57 to 95.83% with an average of 88.51%.

## CONCLUSION

This experiment provides an effective alternative to traditional distillation exercises and instructs students how to manage waste responsibly. Making students accountable for their use of laboratory chemicals emphasizes the need for conservation of materials to minimize waste and exposure to organic solvents.[7] For many instructors, it is difficult to adopt an entirely green curriculum, and this experiment provides a means to move in the direction of greening the organic chemistry laboratory curriculum.[8] The ongoing communal still provides wash acetone throughout the term and represents a constant reminder that proper disposal of waste can lead to effective methods to reduce, reuse, and recycle materials used in the lab. After completing this experiment, students become conscientious of the importance of minimizing waste production for the rest of the term. Introducing this distillation in the undergraduate organic laboratory encourages students to see the connection between rising laboratory fees, the expense of chemical waste disposal, and the ability to take small steps that make a huge difference in reducing hazard exposure, lowering cost, and benefitting from recycling. These are concepts that students retain well beyond their experience in the organic chemistry lab.

## ASSOCIATED CONTENT

### Supporting Information

Student instructions, including background information, prelab questions, distillation procedure, result tables, and student report expectations; instructor notes including answers to the prelab questions, the setup procedure for the communal recycling still, and student results. This material is available via the Internet at http://pubs.acs.org.

## AUTHOR INFORMATION

### Corresponding Author

*E-mail: owenmcdougal@boisestate.edu.

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