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Waterless Condensers for the Teaching Laboratory: An Adaptation of Traditional Glassware

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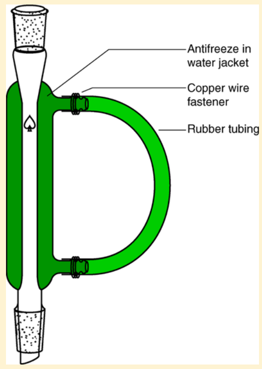
ABSTRACT: A simple adaptation of traditional "chemistry kit" condensers for the organic chemistry teaching laboratory is described. These waterless condensers have been employed safely with most solvents. They can be easily fabricated, stored, and used in the same manner as water-cooled condensers. These condensers were utilized in several different types of reactions and in an ethanol–water distillation providing the same product yields as reactions utilizing traditional water-cooled condensers.



KEYWORDS: *Second-Year Undergraduate, Laboratory Instruction, Organic Chemistry, Laboratory Equipment/Apparatus*

Condensers are common pieces of laboratory equipment that are routinely utilized in the undergraduate organic teaching laboratory for the essential techniques of reflux[1] and distillation.[2] Starting in 1929,[3] modifications of condensers for reflux or distillation have appeared in this *Journal* with the most recent appearing in 2003.[4] Condensers are typically cooled with running tap water at a rate that will maintain a steady flow or employ expensive water recirculation systems. In the hands of inexperienced undergraduates, this can be between 5.5–180 L of water per hour.[5] Water in the public supply is 6.6% of fresh water usage in Indiana.[6] In 2011–2012, Butler University used over 229,000,000 L of potable water. Water flowing through condensers is potable water that is wasted. Reflux conditions with static fluid condensers and a variety of solvents have been tested with a similar system.[7] Herein, an adaptation of condensers contained in traditional teaching kits is described that allows them to function without the need for flowing water.

Typical organic chemistry glassware kits contain both a Leibig or jacketed condenser and a West condenser. The West condenser, with its narrow inner tube, did not work as well in this application when compared to the Leibig condenser with the larger inner diameter. The jacketed condenser was filled with antifreeze concentrate (Peak Long Life full strength concentrate), the hose connections on the condenser were joined by rubber tubing to make a closed system, and the tubing was secured with copper wire (Figure 1).[8] Tygon and Nalgene tubing became too slippery and did not consistently remain attached to the condenser in this application. The condenser was then utilized in a variety of reflux and distillation experiments throughout a year-long organic chemistry laboratory sequence. The expected outcomes of each experiment

Figure 1. Waterless condenser apparatus (19/22), note that air bubbles do not adversely impact performance.

were compared to those observed in previous experimental cycles employing traditional condensers.

FINDINGS

All of the findings are found in Table 1. For a distillation experiment (ethanol from an ethanol/water mixture), the waterless condenser worked as well as a water-cooled condenser producing identical yields and purities (10–15 mL of 80+ proof ethanol or "flammable fractions"), although the condenser was warm to the touch at the end of the laboratory

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Table 1. Results from Organic Laboratory Reactions Where Condensers Were Used

Experiment Title	Time Condenser Used (h)	Solvent Used	Distillation or Reflux	Expected Outcome	Waterless Condenser Result Averages[a]	Estimate of Water Savings per Student (L)[b]
Fermentation of Sugar	1.0	H_2O (75 mL)	Distillation	10−15 mL of concd ethanol solution	12.2 mL	5.54−171.4
S_N2 Displacement of an Alkyl Halide	1.0	EtOH (20 mL)	Reflux	10−65% isolated yield	34% yield	5.54−171.4
Acid Catalyzed Dehydration of a Tertiary Alcohol	0.25	9 M H_2SO_4 (4 mL)/ ROH[c] (3 mL)	Reflux	40−60% product formation by GC−MS	48% conversion to alkene isomers	1.35−42.8
Bromination of *trans*-Cinnamic Acid	0.30	CH_2Cl_2 (20 mL)	Reflux	40−60% isolated yield	60% yield	2.72−85.7
Grignard Reaction	1.5	Et_2O (15 mL)	Reflux	20−60% isolated yield	80% yield[d]	8.26−257.1
Independent Synthesis	1.5	Et_2O (15 mL)	Reflux	10−30% isolated yield	11% yield	8.26−257.1

[a]Allowing for typical student error over 60 trials per experiment. [b]Estimate based upon low and high student water flow rate. [c]ROH used was 2-methyl-4-phenyl-2-butanol. [d]Crude product, not dried.

period. In fact, the expected outcomes for each experiment performed with the waterless condensers were equivalent to those of the previous iterations in past years as well as the control laboratory section using traditional water-cooled condensers. For reactions in which higher-boiling solvents, 60 °C and higher (methanol, ethanol, tetrahydrofuran, etc.), were heated to reflux, the waterless condenser also performed the same as its traditional counterpart. For lower-boiling solvents (such as dichloromethane and diethyl ether), some students saw solvent loss, up to half of the solvent volume.[9] This was successfully addressed by moving round-bottomed flasks slightly above the heating mantles.

The condensers were successfully shared between all the laboratory sections. Over the academic year, these condensers were used seven times by three of 14 laboratory sections. In the infrequent events of tubing disconnection, ethylene glycol (health 1, flammability 1, reactivity 0) was treated as a chemical spill. Each condenser contained less than 20 mL total volume. There were only three disconnection events, two with larger diameter rubber tubing and one with Tygon tubing. For this reason, condensers with smaller diameter tubing connected to larger hose connections along with the securing of the tubes with copper wire are recommended considerations for this adaptation.

CONCLUSIONS

An easy adaptation using existing equipment in a typical glassware kit to a waterless condenser has been demonstrated. This waterless condenser has been utilized successfully in a variety of typical organic laboratory adaptations. This modification resulted in a water savings of at least 2,600 L over the 2012−2013 academic year. Upon full adoption by all the laboratory sections, the water savings could be more than 13,000 L of water per year. Although this is a small percentage of the water use on campus at Butler University, it is a significant step in the right direction.

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Notes

The authors declare no competing financial interest.

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(8) In our hands, heating the closed system described here has not resulted in hoses coming off, possibly due to the air bubble in the system. If this is a concern, a glassware design utilizing a pressure release valve is described in ref 7.
(9) Upon further evaluation, it was found that some voltage controllers were supplying excess energy to the heating mantles and warming the glassware beyond the boiling point of the solvents, even though they were set to the lowest possible setting. This was also seen in ref 7.