Introducing a Novel and Highly Efficient Water-Saving Condenser
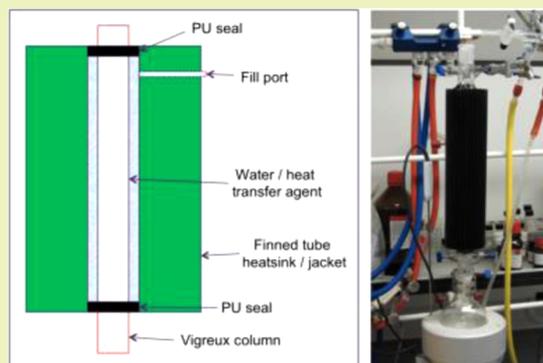
Matthew Grist,*[†,||] Dave Perkins,[†,||] and David M. Barber[‡,||]

[†]AstraZeneca R&D, Mereside, Alderley Park, Cheshire SK11 4TF, United Kingdom

[‡]Chemistry Research Laboratory, 12 Mansfield Road, Oxford OX1 3TA, United Kingdom

S Supporting Information

ABSTRACT: A novel prototype condenser has been constructed utilizing a Vigreux column surrounded by a water jacket that is encased in a finned aluminum tube. The results reported demonstrate the possibility of condensing a variety of solvents at low to medium volumes without the need for a continuous flow of water.

■ INTRODUCTION

The impact of chemistry on the environment is becoming an increasingly important issue to consider when conducting chemical research. The field of green chemistry is ever expanding, developing new and improved reaction protocols that not only reduce energy usage and waste production but can also improve the reaction yield. However, it is the resources that we take for granted that can often be overlooked, for example, the increased energy consumption caused by a raised fume hood sash.[1] This enhanced energy usage not only proliferates the impact chemistry has on CO_2 emissions but also significantly increases the running costs of a chemistry laboratory. While this problem can be controlled with education and by changing behaviors, some problems are not so easy to tackle. One area of wastage that continues to attract heightened attention worldwide is that of water loss. Wasted water needs to be treated in water purification plants before it can be used again, which is highly energy intensive. According to the United States Environmental Protection Agency (EPA), American public water treatment facilities consume nearly 56 billion kWh/year, it is estimated that this equates to enough electricity to power 5 million homes for an entire year. With so much attention from companies and water authorities to reduce water usage, it is surprising that chemists waste countless liters of water on a day-to-day basis. Measurements in our laboratory show that a traditional Leibig reflux condenser can consume between 1 and 4 L of water per minute. Condenser water consumption was measured throughout our laboratory by catching used water, over a timed 1 min period, in a graduated vessel. Therefore, a reaction using a Leibig reflux condenser for 12 h can use between 720 and 2880 L of water. When this figure is considered across the entire chemical research industry

and academia then the energy usage, CO_2 emissions, and the costs become very significant. To this end, we sought to dramatically reduce laboratory water consumption by developing a new reaction condenser that does not require a running water supply. Some laboratories maintain a chilled circulating water supply. We deemed this unsatisfactory as it shifts the energy cost into operation of the chiller system.

The proposed condenser presents many challenges, as it would need to be viable for a wide variety of laboratory solvents under refluxing conditions while also being able to efficiently cool large quantities of solvent, and it would need to consume no water or energy.

Various ingenious methods for reaction solvent condensers that do not require a flow of water already exist. Arguably, the most notable and widely used of these air condensers is the Vigreux column.[2] Usually employed in fractional distillations, Vigreux columns can also be used as an air-cooled condenser for reactions using high boiling point solvents such as toluene. However, Vigreux columns are not applicable for lower boiling point solvents such a dichloromethane and acetone. Also of note is the "Airflux" condenser available from Aldrich.[3] The claims for this condenser are quite modest with use only for solvents with a boiling point over 60 °C, and tandem condensers are often required. The modest performance and relatively high purchase price, £662.60 at time of writing, make this an unattractive option. Our aim is to develop a condenser that can easily out perform an air-cooled Vigreux column and

challenge the efficiency of a Liebig water cooled condenser when using a low boiling point solvent.

Reaction condensers need to be chemically inert to the wide range of reagents and conditions that are at the disposal of a chemist. This naturally made glass is the material of choice for the central column of the new condenser. Surrounding this glass column was placed a finned metal tube (Figure 1). This
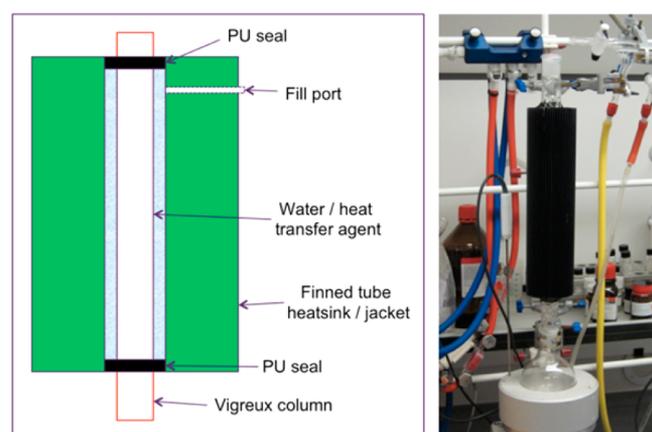

Figure 1. Schematic diagram of the prototype condenser (left) and the prototype (right) being used in the laboratory.

created a very high surface area to dissipate the latent heat energy from the solvent. However, the use of glass connected directly to a metal tube presented us with a problem. The differing thermal expansion rates of the glass and the metal tube would cause the condenser to lose integrity when heated during a reaction.[4]

We postulated that we could solve this problem by using a flexible sealing agent between the glass core and the outer metal tube. Fortunately, the use of a polyurethane adhesive created a sufficient plug to bind the glass core to the metal tube at the top and the bottom of the condenser. The resulting space was then filled with water to act as the heat transfer agent, and our water saving condenser was born. A schematic drawing of our prototype is available in the Supporting Information.

■ RESULTS

To test the efficiency of our condenser, we compared the solvent loss observed with that of a standard straight air cooled condenser and an air-cooled Vigreux column for a variety of low boiling point solvents (Table 1). In each case, a standard B24 jointed air condenser, a B24 jointed air-cooled Vigreux column, and our B24 jointed prototype, all of similar length, were used in a fumehood with a closed sash. We elected to put the condensers under the harshest possible conditions to which they may be subjected in a laboratory and set our heating temperature at 20 °C above the boiling point of the solvent. Heating 50 mL of diethylether at 55 °C with the prototype condenser showed that there was only a 31 mL loss of solvent after 90 min (Table 1, entry 3). This result was significantly superior compared to the complete solvent loss shown by the straight air condenser and the air-cooled Vigreux column after 90 min (entries 1 and 2). With the vigreux column, complete loss of diethylether was observed after 45 min compared to complete loss of with the prototype condenser after 160 min. Dichloromethane was also efficiently retained when used in conjunction with our condenser with only 5 mL being lost after

Table 1. Comparison of Prototype Condenser with Straight Air Condenser and Air-Cooled Vigreux Column Using 50 mL of Solvent

entry[a]	solvent	boiling point (°C)	condenser type	solvent loss (mL)		
				90 time (min)	300 time (min)	960 time (min)
1	Et$_2$O	35	air	50	—	—
2	Et$_2$O	35	Vigreux	50	—	—
3	Et$_2$O	35	prototype	31	50	—
4	CH$_2$Cl$_2$	40	air	9	50	—
5	CH$_2$Cl$_2$	40	Vigreux	5	30	50
6	CH$_2$Cl$_2$	40	prototype	2	2	5
7	Me$_2$CO	57	air	2	19	50
8	Me$_2$CO	57	Vigreux	2	12	30
9	Me$_2$CO	57	prototype	0	2	5
10	MeOH	65	air	3	5	12
11	MeOH	65	Vigreux	0	0	12
12	MeOH	65	prototype	0	2	5
13	THF	66	air	0	9	50
14	THF	66	Vigreux	2	5	50
15	THF	66	prototype	2	2	4

[a]Conducted using 50 mL of solvent in a 100 mL flask heating at 20 °C above the solvent boiling point for the reported time.

960 min at 60 °C (Table 1, entry 6). In comparison, the straight air condenser and air-cooled Vigreux column showed complete loss after 300 and 960 min, respectively (Table 1, entries 4 and 5). Similar results were obtained using acetone (Table 1, entries 7−9), methanol (Table 1, entries 10−12), and tetrahydrofuran (Table 1, entries 13−15), with our prototype condenser consistently out performing the straight air condenser and air-cooled Vigreux column.

However, reactions are often performed on a larger scale than 50 mL. To further compare the efficiency of our prototype against that of an air-cooled Vigreux column, we investigated the solvent loss using 500 mL (Table 2). Fortunately, it consistently demonstrated that it reduced the solvent loss when compared to that exhibited by an air-cooled Vigreux column. Acetone, methanol, tetrahydrofuran, ethyl acetate, and acetonitrile all displayed superior solvent retention when

Table 2. Comparison of Prototype Condenser with an Air-Cooled Vigreux Column Using a 500 mL Scale

entry[a]	solvent	boiling point (°C)	condenser type	solvent loss (mL)
1	Et$_2$O	35	Vigreux	500
2	Et$_2$O	35	prototype	500
3	CH$_2$Cl$_2$	40	Vigreux	500
4	CH$_2$Cl$_2$	40	prototype	240
5	Me$_2$CO	57	Vigreux	40
6	Me$_2$CO	57	prototype	20
7	MeOH	65	Vigreux	40
8	MeOH	65	prototype	<5
9	THF	66	Vigreux	25
10	THF	66	prototype	<5
11	EtOAc	77	Vigreux	35
12	EtOAc	77	prototype	<10
13	MeCN	82	Vigreux	35
14	MeCN	82	prototype	0

[a]Conducted using 500 mL of solvent in a 1 L flask heating at 20 °C above the solvent boiling point for 16 h.

using our condenser after boiling for 960 min (Table 2, entries 5−14).

Of particular note were the results obtained when using dichloromethane. Heating 500 mL of dichloromethane at 60 °C for 960 min using an air-cooled Vigreux column resulted in complete solvent loss (Table 2, entry 3). However, using the prototype under identical conditions only yielded a 240 mL loss of solvent (Table 2, entry 4). Further investigation showed that the DCM loss with the prototype after 16 H was observed in just over 2 H with the air-cooled Vigreux column. The vastly superior result effectively represents how it can be used with large quantities of low boiling point solvents. Despite the success of the prototype when using dichloromethane, it was unfortunately found to be incompatible with diethylether on such a large scale, with complete solvent loss being observed within 960 min at 20 °C above the boiling point (Table 2, entry 2). The substantial difference in the results obtained between diethylether and dichloromethane show the limitation of the condenser on 500 mL. The results for higher boiling point solvents such as acetone and methanol show that the prototype provides adequate cooling for 500 mL of solvent with a lower solvent loss than that shown by the air-cooled Vigreux column.

To further test the limitation of the prototype, a series of experiments on 1 L of solvent were performed for the lower boiling point solvents (Table 3). At this volume, the prototype

Table 3. Comparison of Prototype and Vigreux Column on a 1 L Scale

entry[a]	solvent	boiling point (°C)	time (min)	condenser type	solvent loss (mL)
1	CH_2Cl_2	40	195	Vigreux	990
2	CH_2Cl_2	40	360	prototype	970
3	Me_2CO	57	320	Vigreux	966
4	Me_2CO	57	360	prototype	570
5	MeOH	65	360	Vigreux	780
6	MeOH	65	360	prototype	160
7	THF	66	160	Vigreux	991
8	THF	66	360	prototype	180
9	EtOAc	77	240	Vigreux	987
10	EtOAc	77	360	prototype	100
11	MeCN	82	360	Vigreux	510
12	MeCN	82	360	prototype	40

[a]Conducted using 1 L of solvent in a 2 L flask heating at 20 °C above the solvent boiling point for the reported time.

struggled to retain dichloromethane, with a loss of 960 mL in 360 min (Table 3, entry 2). However, when compared to an air-cooled Vigreux column (Table 3, entry 1), our condenser dramatically increased the time that the solvent could be refluxed. The employment of methanol showed that the prototype could greatly reduce solvent loss after heating at 85 °C for 360 min, with it only losing 160 mL (Table 3, entry 5) compared to 780 mL loss for the air-cooled Vigreux column (Table 3, entry 6). Exceptional results were also obtained when using tetrahydrofuran (Table 3, entries 7−8) and ethyl acetate (Table 3, entries 9−10). On both occasions, the prototype retained between 80% and 90% of the starting solvent volume, whereas the air-cooled Vigreux column showed almost complete loss in a reduced time frame. As the boiling point of the solvent increases still further, lower solvent losses are observed with the prototype condenser.

The new condenser has other additional benefits over traditional water condensers. It is very easy to use as it has no water wheels, tubing, or connectors, all of which can fail leading to water leaks and the associated mess. To identify leaks from water condensers, overnight water monitors can be used to notify night stewards of water issues. Again, the use of the new condenser negates the requirement for these control measures. Set-up is as simple as just clamping the condenser in position over the reaction. Even the need for clamping is a precautionary measure rather than a necessity; due to its symmetrical design, the center of gravity is straight down the center of the condenser.

CONCLUSIONS

In conclusion, we believe that we have developed a novel replacement for the traditional water condenser that can be of enormous benefit in reducing water usage. We appreciate that some modern laboratories are equipped with recirculating chilled water systems to reduce water usage. These systems consume significant amounts of energy in maintaining the chilled temperature and so still present a signifcant environmental impact. Measurements in our laboratory of a chiller unit used for a single condenser show an energy consumption of 10.6−15 kWh/day depending on the solvent being used.

Our findings indicate that this prototype can be used routinely for all low volumes of regularly used solvents being heated overnight. Solvent losses increase as solvent volumes increase, but the prototype still remains acceptable for a wide variety of solvents at larger volumes.

Importantly, our trials have set the heating at a very challenging 20 °C above the solvent boiling point. The aim of this was to set a harsh test of the equipment. By lowering this differential to 5 °C, experiments indicate that we can reduce solvent losses still further so all solvents up to 1000 mL volumes are retained, including low boiling solvents such as DCM and ether. The results obtained have shown that the newly developed condenser is a viable replacement for a Liebig condenser. Coupled with the ease of set up, the absence of water leakage issues, and the energy-free and cost-free use, we believe this prototype condenser should have a huge future within the laboratory environment.

ASSOCIATED CONTENT

ⓈSupporting Information

Construction of our prototype. This material is available free of charge via the Internet at http://pubs.acs.org.

AUTHOR INFORMATION

Corresponding Author

*E-mail: MatthewGrist@astrazeneca.com.

Author Contributions

‖The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. These authors contributed equally.

Notes

The authors declare no competing financial interest.

ABBREVIATIONS

°C, degrees centigrade
CO_2, carbon dioxide
DCM, CH_2Cl_2 − dichloromethane
EPA, United States Environmental Protection Agency
ether, diethyl ether
EtOAc, ethyl acetate
h, hours
kWh, kilowatt hour
L, liters
MeCN, acetonitrile
Me_2CO, acetone
MeOH, methanol
min, minutes
mL, milliliters
PU, polyurethane
THF, tetrahydrofuran

REFERENCES

(1) Marian, J. S., Jr. A Study of Factors Affecting Fume Hood Energy Consumption, American Auto-Matrix: Exort, PA, September 2009.
(2) Vigreux, H. Annales de Chemie Analytique et de Chemie Applique et Revue de Chemie Analytique Reunies, 1919, Vol. *1*, p 211.
(3) Sigma Aldrich Airflux condenser. http://www.sigmaaldrich.com/labware/labware-products.html?TablePage=13451205 (accessed May, 3, 2013).
(4) Thermal expansion coefficients were obtained from Wikipedia: Aluminium 23.1×10^{-6} m/m K, borosilicate glass $\sim 3 \times 10^{-6}$ m/m K, and water 69×10^{-6} m/m K at the time of drafting. http://en.wikipedia.org/wiki/Borosilicate_glass, http://en.wikipedia.org/wiki/Aluminium, http://en.wikipedia.org/wiki/Thermal_expansion_coefficient (accessed May 3, 2013).