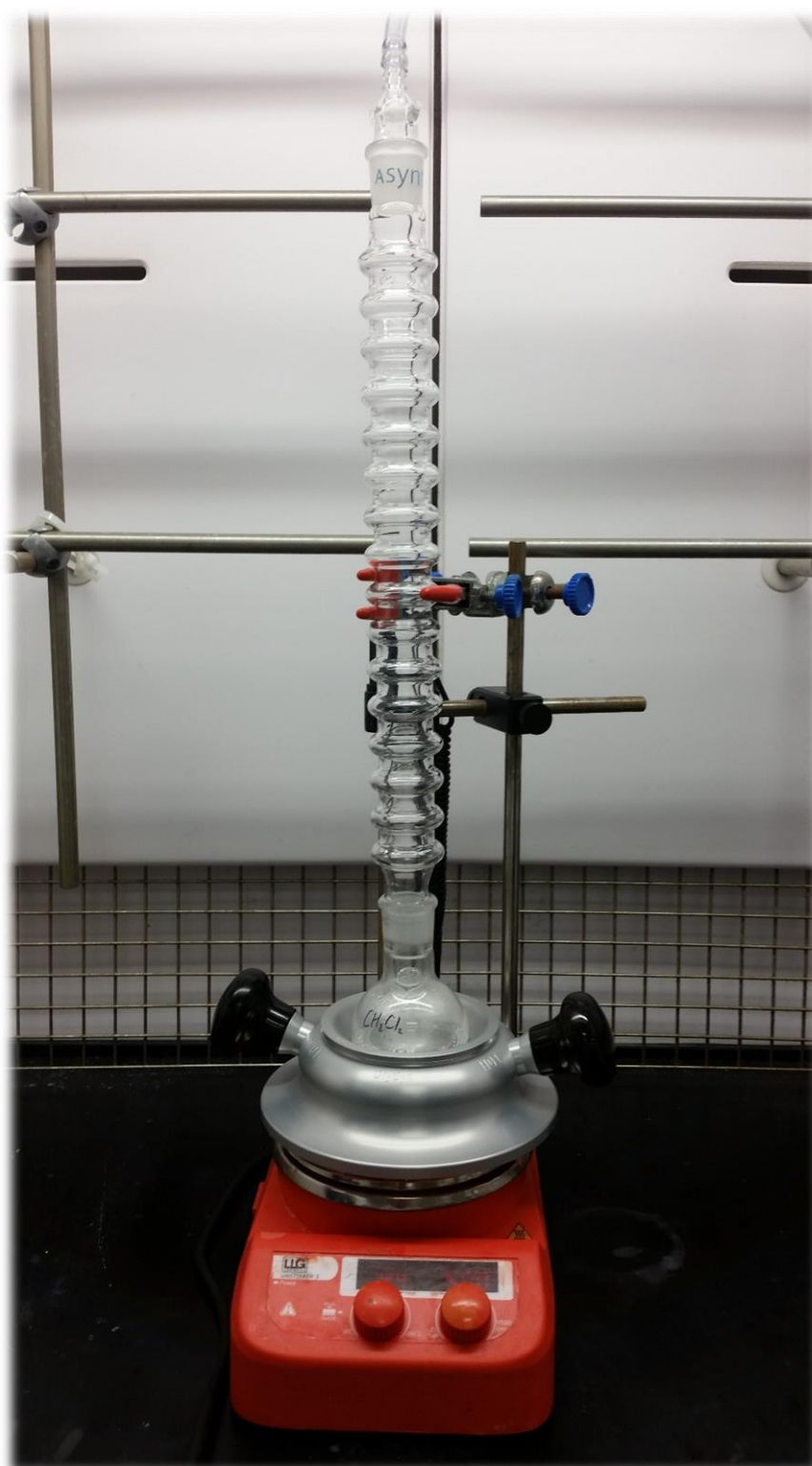


Testing the performance of Asynt CondensSyn Waterless Condensers under “real” laboratory conditions



Dr Nicholas White, June 2016

Introduction:

Synthetic chemists often need to perform reactions at elevated temperatures in refluxing solvent – traditionally this has been achieved by heating the reaction mixture in a bath of silicone oil and using a water-driven condenser to cool the hot solvent and prevent it evaporating. There are significant disadvantages to both the use of silicone oil and water condensers, although more modern laboratory equipment can overcome both of these.

Silicone oil is expensive (typically > AU\$100/L) and is difficult to clean up when spilled due to its insolubility in water and low solubility in common organic solvents used for cleaning (acetone, alcohols).

The use of water-driven condensers present several problems, firstly that a large amount of water is used in this process, which is wasteful and potentially expensive. For example, each of the tests using a water-driven condenser in this report used approximately 1,400 litres of water (a flow rate of 1.0 L/minute for 24 hours). Furthermore, the additional clutter associated with attaching the condensers both to the reaction set-up, and to the water supply and drainage can cause difficulties in terms of condenser tubes that tangle with gas supplies and clamps. Finally, the possibility of leaks from the condenser supply can result in flooding and may be dangerous if a water condenser is used with a reagent that reacts violently with water (such as an organometallic species).

The use of silicone oil can be avoided by using solid metal heating blocks such as Asynt DrySyn systems and Radley heat-on blocks. This avoids the need to purchase expensive silicone oil, and means there is no chance of spilling oil. More recently, the development of water-free condensers has offered the potential for even simpler laboratory chemistry, although until very recently, these have typically been very large (and heavy), making them relatively impractical. In the last year, Asynt have released an operationally-simple, lightweight solventless condenser known as the CondenSyn – this report examines the performance of this under “real-world” laboratory conditions, and compares it against water-driven condensers.

Testing:

It was decided to test the CondenSyn under relatively tough conditions, as close as possible to those used in a typical laboratory synthesis. A 100 mL round bottomed flask containing 50 mL of a given solvent was heated to a temperature 10% higher than the stated boiling point of the solvent for 24 hours under bubbling nitrogen gas (see Figure 1). As the tests were conducted in Canberra, at an altitude of approximately 580 metres above sea level, this is significantly above the “real” boiling point of the solvent, and was sufficient to ensure vigorous boiling for all solvents tested.

The CondenSyn and water-driven condensers were tested “side-by-side” in the same fumehood using identical heater-stirrers and DrySyn heating blocks, with the same temperature and stirring settings to ensure the fairest possible comparison. Using this side-by-side set-up allowed us to control for variation in laboratory temperature and fumehood airflow. After 24 hours heating and stirring, the mass of the flask and solvent was measured and the amount of solvent lost calculated.

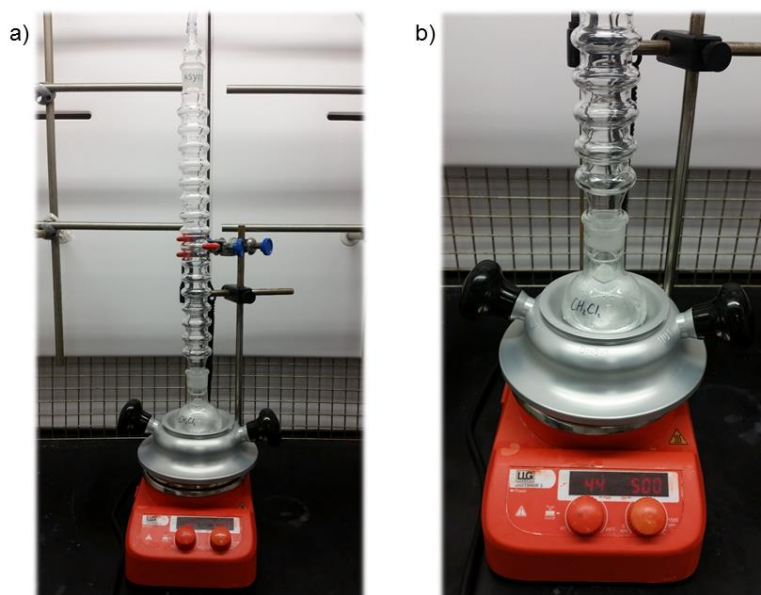


Figure 1. Photographs of DrySyn/CondensSyn set-up showing refluxing dichloromethane: a) entire set-up including nitrogen gas supply; b) close-up photograph showing heating/stirring settings and refluxing solvent.

The CondensSyn was tested against two conventional water-driven condensers – a Davies double surface condenser (which contains both an inner and outer water jacket), and a coiled condenser (Figure 2). It should be noted that both of these are more efficient (and more expensive) than a standard single surface Liebig condenser. Both are approximately 30 cm in length, while the CondensSyn is slightly longer (just over 40 cm). While the CondensSyn is longer than the water-driven condensers and so may gain an advantage from its length, we found that the vast majority of water-driven condensers in our department were 30 cm (or shorter), and so this was a representative “real-world” test of the CondensSyn against what would normally be used in our facility.



Figure 2. Photographs of condensers used in this test: a Davies double surface condenser (left), a coiled condenser (centre) and a CondensSyn (right). A 40 cm ruler (far right) is shown to give an estimate of scale.

Initially, the low-boiling solvent dichloromethane was used, as it was felt that this would be a difficult solvent to condense and so be a stern test for any condenser. This was repeated in triplicate for the CondensSyn, and both water-driven condensers were also tested. The results are presented in Table 1, and show that the CondensSyn substantially out-performed either of the water-driven condensers.

Table 1. Percent of dichloromethane lost during 24 hours refluxing under bubbling nitrogen gas using various condensers for cooling.

Condenser	% Dichloromethane lost
CondensSyn	6.0, 3.5, 7.0 (<i>mean: 5.5</i>)
Davies double surface condenser	11
Coiled condenser	13

The relatively small amount of solvent lost using the CondensSyn is remarkable given the robust heating and bubbling nitrogen (which will increase evaporation), and the small difference between the laboratory temperature (21–24 °C) and the boiling point of the solvent. There is significant variation in our results, which is probably due to a range of variables that are encountered in a laboratory operating under “normal” conditions, specifically changes in ambient temperature, and the airflow through the fumehood (which would have been affected based on how often the fumehood was used during the day).

Having tested the CondensSyn’s performance with a low-boiling solvent (dichloromethane), we next tested the CondensSyn using solvents covering the typical range of temperatures used in a normal synthetic laboratory. Specifically, we used solvents with moderate (tetrahydrofuran), high (toluene) and very high (bromobenzene) boiling points. Given that the Davies double surface condenser performed better than the coiled condenser, we used this as our control in subsequent studies. In all cases, the amount of solvent lost by either the water-driven condenser or CondensSyn was minimal, although with higher boiling solvents, the water-driven condenser performed marginally better than the CondensSyn (Table 2).

Table 2. Percent of solvent lost during 24 hours refluxing under bubbling nitrogen gas using either a CondensSyn or water-driven Davies double surface condenser for cooling.

Solvent	Boiling point ^a	% Solvent lost by CondensSyn	% Solvent lost by water-driven condenser
Tetrahydrofuran	66 °C	1.5	1.0
Toluene	111 °C	0.5	< 0.5
Bromobenzene	156 °C	< 0.5	< 0.5

^aThis is the manufacturer’s stated boiling point for the solvent, although the true boiling point at the altitude the tests were conducted will be less than this. The heater-stirrer was set to a value 10% higher than this, resulting in a vigorous reflux.

Conclusions

The CondensSyn condenser was tested against highly-efficient water-driven condensers in “real-world” laboratory conditions. The CondensSyn displays comparable performance to even the most efficient water-driven condensers available to us. In fact, the CondensSyn displays superior performance with the low-boiling solvent dichloromethane. While it is slightly less effective with moderate and high-boiling solvents, this is not a significant problem, as a negligible volume of solvent is lost after even prolonged heating in these systems.

Given the other advantages of the CondensSyn such as vastly reduced water consumption, less cluttered working environments and ease of setting up and cleaning up reactions, we suggest that the CondensSyn is a superior choice to water-driven condensers. By combining the CondensSyn with a heating block system, reflux set-ups can be prepared quickly and easily with no chance of water or oil spillages.

Technical details

The laboratory temperature was observed to be between 21 and 24°C throughout the testing period. Heating and stirring was conducted using a LIG UniStirrer 3 heater-stirrer with temperature probe and a DrySyn Scholar heating block. A 100 mL B24 round-bottomed flask was loaded with 50±1 mL of accurately-weighed solvent, and the temperature probe set to a temperature 10% higher than the stated boiling point of the solvent (*i.e.* 44 °C for dichloromethane, 73 °C for tetrahydrofuran, 122 °C for toluene, and 172 °C for bromobenzene). The refluxing solvent was stirred at 500 rpm using an 8 mm oval stirring bar. A CondenSyn condenser or water-driven condenser (with a B24 quickfit joint in all cases) was attached to the flask, and a nitrogen bubbler running at approximately 5 bubbles per second (*i.e.* a relatively high nitrogen flow) was added to the top of the condenser (no grease was used on any joints). For the water-driven condenser, a water flow rate of approximately 1.0 L/minute was used, and the tap water temperature was found to be 12–13 °C.

Percentage solvent losses are given to the nearest 1% for values ≥10% and the nearest 0.5% for values <10%.

Conflict of interest statement

The author was provided with a discount on the purchase price of CondenSyns in return for conducting this testing.