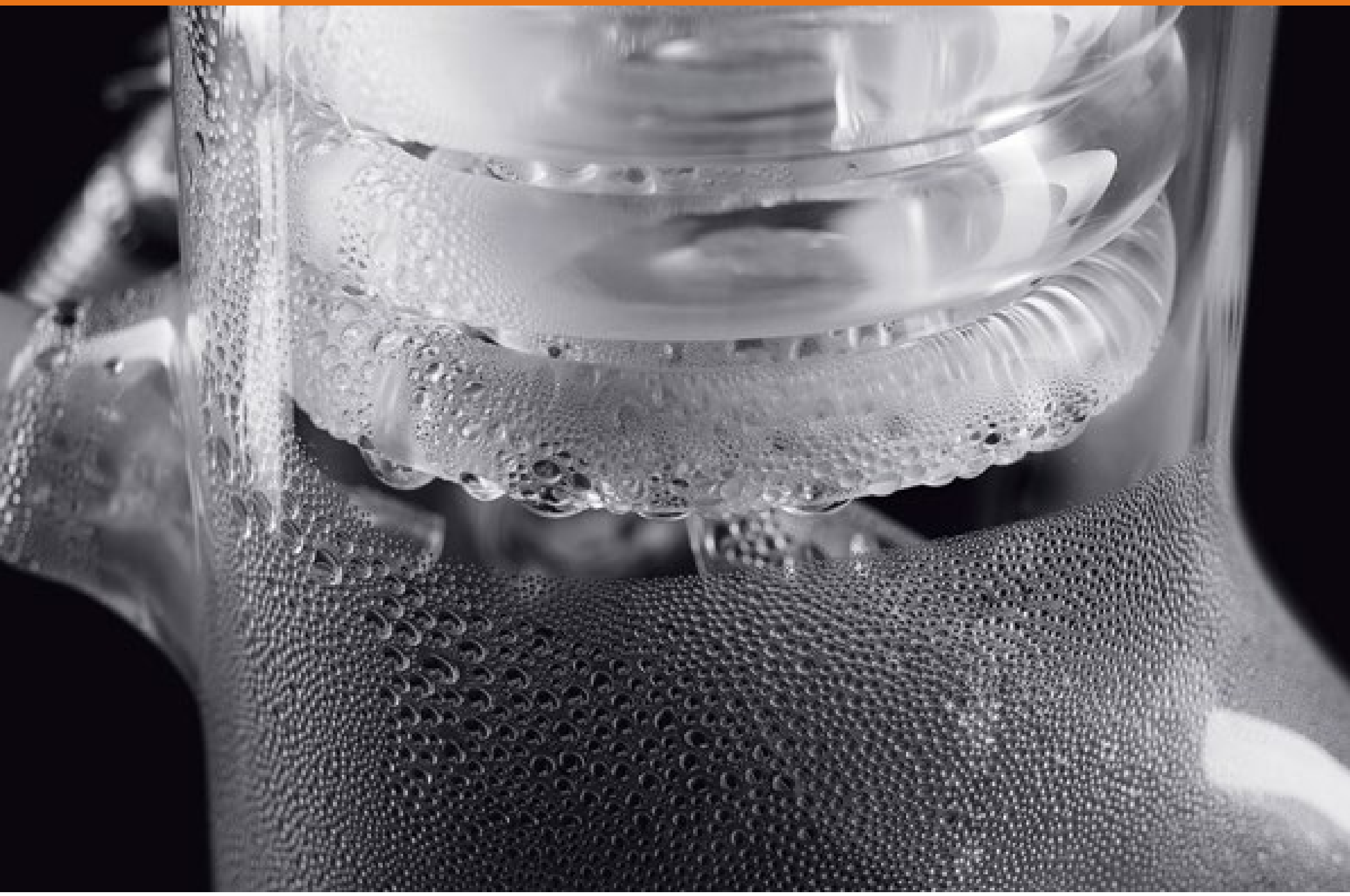
Dry Ice Condenser vs. Recirculating Chiller

Category: Rotary Evaporator
Whitepaper published July 2020



heidolph
research made easy

Comparison of a dry ice condenser with a high-performance condenser with cooling coils and recirculating chiller

Choosing the condenser for an evaporator system – a key decision . The choice of the glass condenser is one of the fundamental decisions in the purchase of a new rotary evaporator system. The broad spectrum of available glass condensers can roughly be separated in two main sections: dry ice condensers and condensers with cooling coils, which work with a tap water connection or in combination with a recirculating chiller. Both variants bear their pros and cons that have to be considered for the purchase decision. This whitepaper compares a Heidolph G5 dry ice condenser to a Heidolph G3 XL cooling coil condenser in combination with a recirculating chiller regarding their handling and performance.

Reasons and beliefs for using dry ice condensers

Many people tend to choose the dry ice condenser over the ones with cooling coils because of the following reasons: To work with a dry ice condenser, neither the purchase of additional equipment like a recirculating chiller, nor the availability of a tap water connection is required. Therefore, the evaporator system takes up less space compared to a system with a recirculating chiller and omits the necessity of tubes for the transport of the cooling liquid, no matter if tap water or a coolant is used. Furthermore, the use of a dry ice condenser does not induce a primary energy consumption like running a recirculating chiller.

One of the main beliefs behind the purchase of a dry ice condenser is that the colder is the better – which is, in fact, not true. The high temperature differences that are present in an evaporator system with a dry ice condenser put the glassware under a high stress. If the hot steam from a solvent hits the surface that is cooled to –78 °C by a mixture of dry ice and isopropanol or acetone, the glass is faced with a temperature difference of 118 °C, assuming that the heating bath temperature is 40 °C. This stress on the glass, although glass condensers are manufactured from highly stress resistant borosilicate glass, leads to a notably higher tendency of glass breakage during the work with a dry ice condenser.

While being of benefit for very low boiling solvents like diethyl ether, the very low temperature present in a dry ice condenser can be of disadvantage if a higher boiling solvent shall be evaporated due to the tendency of high boilers to condensate too early, before reaching the condenser surface. A performant use of a dry ice condenser with solvents like ethanol or water, that bear a high heat of vaporization is almost impossible.

What should also be considered is that the unattended operation over several hours or even overnight is not possible with a dry ice condenser, because it requires a constant refill with dry ice to keep its temperature and performance. If the evaporation process with a dry ice condenser is left unsupervised over a longer period of time, the danger of the solvent used for the cooling mixture getting hot and starting to evaporate into the ambience air is given. This can lead to intoxication and ignition risks. Dry ice has to be handled carefully anyway, otherwise the risks of skin burns and suffocation are given. It is mandatory to use protection gloves when working with dry ice. Additionally, the cooling mixture has to be prepared per use every time. Because there is a severe reaction between the dry ice and the solvent, the condenser cannot be filled at once, so this must happen step by step to avoid spillage of the solvent that is used for the cooling mixture.

The dry ice condenser holds up to 800 g dry ice per filling, which has to be taken into consideration when looking at the acquisition costs of the system: The dry ice sublimates during the process, so it cannot be re-used. The solvent needed for the mixture is mostly disposed because it has a tendency to absorb ambience moisture which leads to a loss of performance after a few cycles. Dry ice and solvents cause running costs when working with such a system.

Condensers with cooling coils provide a broader usage spectrum

Compared to the dry ice condensers, condensers with cooling coils, especially high-performance models that provide a large cooling surface, can be used with a broad spectrum of solvents. The combination with a recirculating chiller allows a precise temperature control that can be adjusted to the process. The large variety of recirculating chillers to choose from, ranging from lower power levels like 250 Watts to high-performance ones like 1200 Watts, allows picking the right model specifically needed for the process that will be run with the evaporator system.

This creates two noteworthy benefits: Glass breakage of a condenser with cooling coil due to high temperature differences is very unlikely. Condensers with cooling coils normally run at a temperature between 15 – 5 °C. The temperature difference the glass is facing is lowered to 25 – 35 °C, which the glass can easily withstand. In addition, the evaporation of low boiling solvents like diethyl ether becomes as performant as the evaporation of very high boiling solvents like DMSO, because the cooling temperature can be adjusted to the process and optimal workload of the glass condenser.

The evaporator system with a recirculating chiller can also be left unattended over long periods of times, without loss of performance or creating safety risks. The handling is very easy – once set up it only has to be switched on and off again. Some evaporator systems even offer the possibility to control the temperature of the recirculating chiller over the evaporator’s control panel.

After the purchase and installation the operating costs only refer to the energy consumption of the system with a recirculating chiller. Therefore, it should be preferred towards a setup where tap water is wasted. The cooling liquid can be used over a long period, since it is enclosed in a cooling cycle and mostly contains substances to prevent the formation of algae.

Objective: Comparison of a dry ice condenser and a high-performance condenser with cooling coils in combination with a recirculating chiller

Heidolph has compared two options of glass condensers regarding their handling and performance. A Hei-VAP Expert Control evaporator system with a Rotavac vario control rpm regulated vacuum pump was equipped with a G5 dry ice condenser and a G3 XL high-performance condenser with cooling coils in combination with a Hei-CHILL 350 recirculating chiller.

Method: Performance and handling comparison of a Heidolph G5 dry ice condenser and a Heidolph XL cooling coil condenser

The G5 dry ice glass set was installed as designated on the Hei-VAP Expert Control evaporator system using the provided silicone ring for sealing to achieve the best possible system tightness. The condenser was supplied with a cooling mixture of dry ice and isopropanol, temperature 78 °C. After every experiment, the dry ice was refilled.

The G3 XL glass set with cooling coils was installed as designated on the Hei-VAP Expert Control evaporator system and connected to a Hei-CHILL 350 recirculating chiller, filled with 20 l of Kryo 30 cooling liquid. The temperature of the recirculating chiller was set to 5 °C.

The evaporation times have been determined in the following manner: The heating bath was filled with water until the second fill line and preheated to 50 °C. The values for rotation and vacuum were set according to table 1. The rotation was started. After it reached the set value, the vacuum was started.

10 mb before the set value, the flask was put into the heating bath and the time measurement with a stop watch was started. The measurement was stopped after there was no solvent left in the flask. (characteristic: towards the end of the process a clearly visible ring of solvent forms inside the flask which then fades. Time is stopped after the fading of the ring).

Solvent		**Set heating bath temperature**	**Set rotation speed**	**Set vacuum value**
Water	**50 ml**	50 °C	140 rpm	20 mbar
Water	**250 ml**	50 °C	140 rpm	20 mbar
Ethanol	**50 ml**	50 °C	140 rpm	60 mbar
Ethanol	**250 ml**	50 °C	140 rpm	60 mbar

Tab. 1: Parameters for the determination of the evaporation times

A: Experiments with water

The evaporation of 50 ml water takes 06:30 min. While the pump was not contaminated, some of the water formed into ice crystals on the condenser because of the cold temperature (Fig. 1). Because of that ice formation, only 32 ml from the 50 ml could be recovered.

Fig. 1: Ice crystals form inside the G5 condenser

6 min after starting the evaporation of 250 ml water about half of the dry ice has already sublimated. After 14 min the vacuum starts to deviate and after 16 min all of the dry ice has sublimated. Shortly after the whole condenser is steamed up completely. (Fig. 2), the heat is not transported anymore. The pressure rises continuously and the process stagnates. Condensate forms inside the tubing (Fig. 3), indicating that the cooling capacity is not sufficient. The condenser gets so hot that the isopropanol starts to reflux, therefore the process was stopped after 30 min. 5 ml water were inside the pump, 15 were left in the flask. 230 ml were recovered.

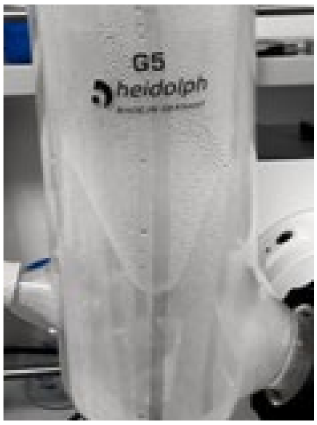


Fig. 2: Completely steamed G5 condenser after 17 min water evaporation

Fig. 3: Condensate inside the tubes during water evaporation with G5 dry ice condenser

Parameters	Time G3 XL	Time G5
50 ml water 50 °C 140 rpm 20 mbar	**06:20 min**	**06:30 min**
250 ml water 50 °C 140 rpm 20 mbar	**21:30 min**	**cancelled after 30 min**

Tab. 2: G3 XL cooling coil condenser vs. G5 dry ice condenser; evaporation times of water

B: Experiments with ethanol

The evaporation of 50 ml water takes 06:30 min. While the pump was not contaminated, some of the water formed into ice crystals on the condenser because of the cold temperature (Fig. 1). Because of that ice formation, only 32 ml from the 50 ml could be recovered.

6 min after starting the evaporation of 250 ml water about half of the dry ice has already sublimated. After 14 min the vacuum starts to deviate and after 16 min all of the dry ice has sublimated. Shortly after the whole condenser is steamed up completely. (Fig. 2), the heat is not transported anymore. The pressure rises continuously and the process stagnates. Condensate forms inside the tubing (Fig. 3), indicating that the cooling capacity is not sufficient. The condenser gets so hot that the isopropanol starts to reflux, therefore the process was stopped after 30 min. 5 ml water were inside the pump, 15 were left in the flask. 230 ml were recovered.

When working with ethanol, there is no notable difference in evaporation times and recovered solvent between the two condensers. Tab. 3 sums up the results of the experiments with ethanol.

Parameters	Time G3 XL	Time G5
50 ml EtOH 50 °C 140 rpm 60 mbar	**03:15 min**	**03:15 min**
250 ml EtOH 50 °C 140 rpm 60 mbar	**11:00 min**	**11:00 min**

Tab. 3: G3 XL cooling coil condenser vs. G5 dry ice condenser; evaporation times of ethanol

C: Discussion of the results and the handling

Concerning the performance, the G5 dry ice condenser equals the G3 XL cooling coil condenser for small volumes and volatile solvents. A problem is finding the right process parameters, because it is not possible to see if the capacity is optimally used. The G3 XL cooling coil condenser provides a marking on the condenser that shows when the optimal workload is reached. For heavy solvents, the G5 dry ice condenser is not suitable: it already fails to condense 250 ml of water if the user does not want to add dry ice constantly to keep the process running. The G3 XL cooling coil condenser system is usable for a broad spectrum of solvents and is not limited to a narrow range of boiling points. It can be even used unattended over several hours or overnight.

During the running process it was observed that the dry ice reacts severely inside the G5 condenser. The more load the condenser is put under, the faster the dry ice sublimates which causes a strong bubbling. If the condenser is fully packed with dry ice it sometimes happens that the solvent runs out on top of the condenser and runs down along the surface.

Under heavy load the condenser has to be refilled continuously to maintain the cooling capacity. If that refill does not happen in time, it comes to a fast heat up of the condenser which leads to a reflux of the solvent inside which is released as gas into the surrounding. This can be dangerous.

Compared to that, the G3 XL cooling coil condenser does not require any further maintenance after being switched on and set to the desired temperature.

Because of the high temperature difference towards the ambience the G5 dry ice condenser is constantly coated with condensed air moisture which forms drops that run down on the condenser and drop onto the surface on which the evaporator stands. If the setting of the recirculating chiller is correct, there is no formation of condensate at the G3 XL cooling coil condenser.

If the evaporator system is left open between two processes, meaning, if no rotation flask is attached, ice crystals from air moisture form inside the G5 dry ice condenser (Fig. 4). This can lead to contamination of the product with water when the next evaporation process is carried out. This formation of ice does not occur at the G3 XL cooling coil condenser.

Fig. 4: Ice crystals from air moisture forme inside the G5 condenser between processes

Conclusion: High-performance condensers with cooling coils allow working with a broader spectrum of solvents

These tests and observations indicate that dry ice condensers provide good performance when working with small amounts of solvents with low to medium boiling points. For large amounts of solvents or those with higher boiling points, the work with a dry ice condenser becomes tiring, because it requires constant maintenance to keep the process running.

Concerning the easier handling and the possibility to evaporate a broad variety of solvents at high rates, a system with a high-performance condenser with cooling coils and a recirculating chiller, chosen specifically to fit the later purpose, covers more applications with less need of surveillance and maintenance.

For any technical questions, application support etc. please contact us under:

EUROPE / AFRICA / ASIA
ASIA PACIFIC / SOUTH AMERICA

Heidolph Instruments GmbH & Co. KG
Walpersdorfer Str. 12
D-91126 Schwabach

Phone: +49 9122 9920-0
Fax: +49 9122 9920-0

sales@heidolph.de
www.heidolph.com

NORTH AMERICA

Heidolph North America
1235 N Mittel Blvd. Suite B
Wood Dale IL 60191

Phone: +1 224-265-9600
Fax: +1 224 265-9611

custserv@heidolph.com
www.heidolphna.com

heidolph
research made easy