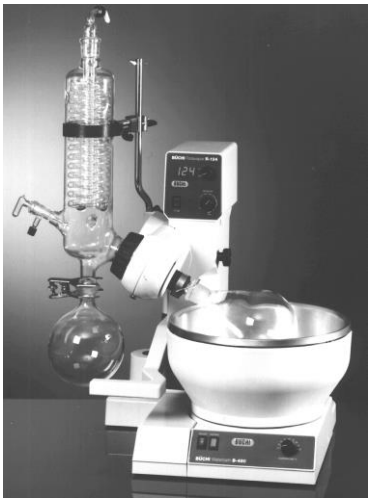
SOP: BÜCHI ROTARY EVAPORATOR

BÜCHI ROTARY EVAPORATORS - ROTAVAP

Rotavaps are used for:

- Distillation of solvent
- Concentration of solutions and suspensions
- Crystallization or recrystallization
- Synthesis and purifying of fine chemicals
- Soxhlet extraction
- Powder and granules drying
- Recycling of solvent

There are different kinds of glass assembly systems in the Büchi range. The ones predominantly used in the Chemistry Department are the A or the V glass assembly systems.



A GLASS ASSEMBLY

The diagonal condenser is very well suited for simple distillations at a high rate of throughput. It is the low-priced model but requires more room.

Suitable for:

- Distillation of solvent
- Concentration of solutions and suspensions
- Drying of powder and granules
- Starting of crystallization processes

V GLASS ASSEMBLY

The vertical design saves space. Glass assembly V is very well suited for distillations of simple and high-boiling point solvents.

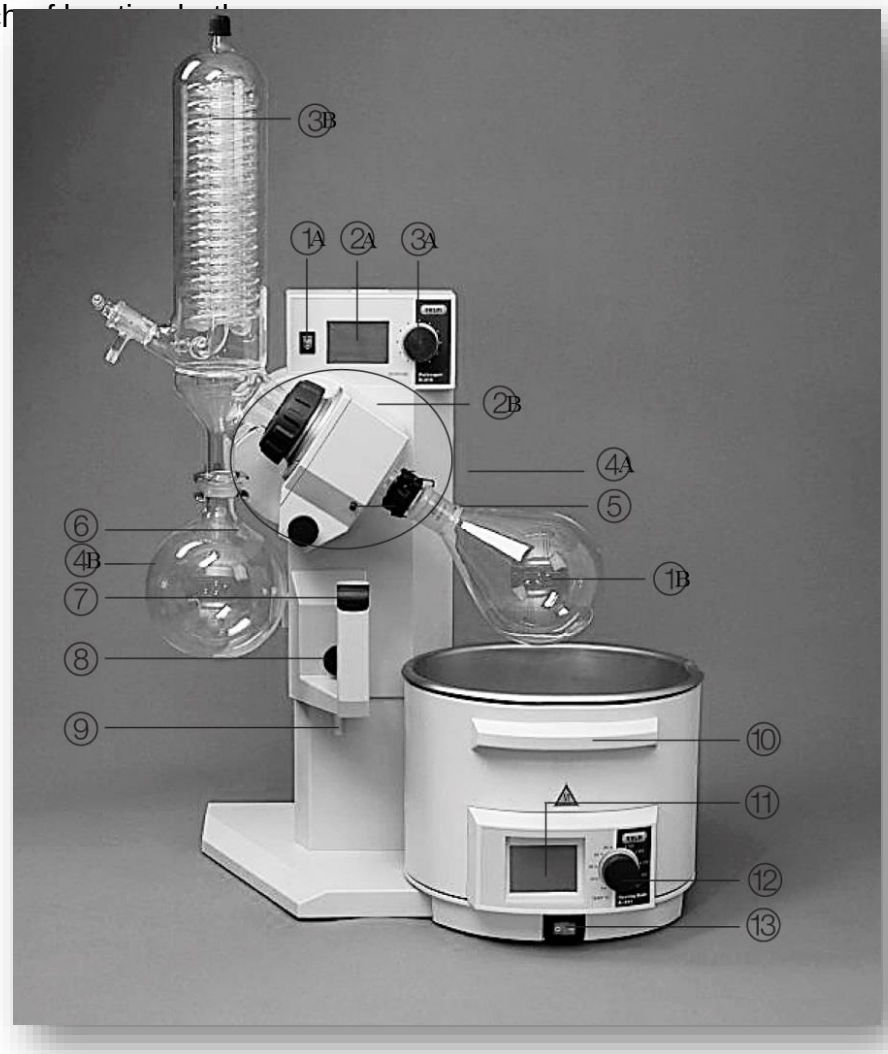
Suitable for:

- Distillation of solvent
- Concentration of solutions and suspensions
- Drying of powder and granules
- Starting of crystallization processes

EVAPORATOR PARTS

1B - Evaporation area
2B - Rotation drive including vapour duct
3B - Cooling area
4B - Receiving flask

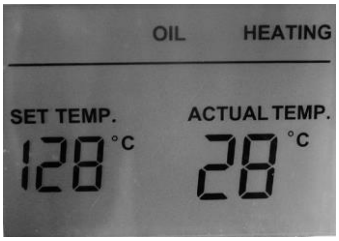
1A - Mains switch of Rotavapor
2A - Display for rotation speed and vapour temperature (only on R-215)
3A - Adjusting knob for rotation speed
4A - Combi Clip for easy flask/vapour duct removal
5 - Lock button to block the drive unit
6 - Knob for immersion angle adjustment
7 - Quick-action jack to lower and raise the evaporating flask
8 - Knob for end stop adjustment
9 - End stop detection
10 - Heating bath handle
11 - Display of bath temperature
12 - Adjusting knob for setting the bath temperature and selecting between water or oil bath operation
13 - Mains switch of heating bath



PROCEDURES FOR CORRECT USE

Setting the heating bath temperature

In water bath mode, the heating bath can be operated with both water or oil. A maximum setting up to 95 °C is possible in water bath mode and 180^0C in oil bath mode.

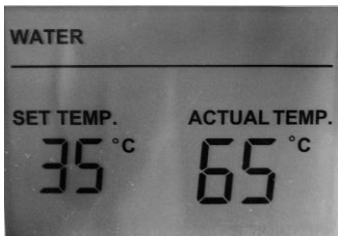


To switch the heating bath from water bath mode to oil bath mode, proceed as follows:

a) Switch off the heating bath.
b) Turn the adjusting knob to any position except for 0°C or 180°C.
c) Switch on the heating bath.
d) As soon as the temperature is indicated on the display, turn the knob to the 180°C position within 3 seconds. The indication “OIL” will now appear on the display.

To switch the heating bath from oil bath mode to water bath mode, proceed as follows:

a) Switch off the heating bath.
b) Turn the adjusting knob to any position except for 0°C or 180°C.
c) Switch on the heating bath.
d) As soon as the temperature is indicated on the display, turn the knob to the 0°C position within 3 seconds. The indication “WATER” will now appear on the display.



Selecting the set temperature

With this setting you can ensure, that the heating bath temperature cannot be changed during the distillation process.
To carry out the setting, proceed as follows:

a) Switch off the heating bath.
b) Turn the adjusting knob to the 180°C / 95°C (max) position.
c) Switch on the heating bath. The set temperature setting flashes in the display.
d) Turn the knob within 10 seconds to the desired set temperature, e.g. 60°C and wait until the set temperature setting stops flashing.

This temperature is now kept whenever the heating bath is switched on and cannot be changed with the adjusting knob anymore.

Changing/switching off the set temperature

To change or switch off the set temperature, proceed as follows:

a) Switch off the heating bath.
b) Turn the adjusting knob to the 0°C (min) position.
c) Switch on the heating bath. The set temperature setting is now deleted and the temperature can be selected via the knob again.

Immersion angle of the evaporating flask into the heating bath

The immersion angle into the heating bath is, by default, set to 30°. If you need to operate with another angle, e.g. when operating with a small flask, the angle can be changed as follows:

a) Turn off the instrument.
b) Hold the glass assembly with the one hand and loosen the anchoring with the other hand by pulling the knob.
c) Set the condenser in the desired position by tilting the drive unit accordingly and let it catch.
d) Release the knob.

Lowering and raising the evaporating flask into and out of the heating bath

The stop-position of the quick-action jack can be adjusted so that neither the vapour tube, the manifold, or the Combi Clip touch the bottom or edge of the bath and a desired immersion depth of the evaporation flask in the heating bath is achieved.

To adjust the stop-position of the quick-action jack, proceed as follows:

a) Push the button (1) up or down to move the quick-action jack to the desired position.
b) Pull the knob (2) and release it again. The stop-position (3) is now fully extended and will remain in this position until adjusted again.
c) To re-adjust the stop-position later, push the button (1) up to raise the quick-action jack a little, then proceed as described above.

Selecting the distillation conditions

To achieve optimal distillation conditions, the distillation energy supplied by the heating bath must be removed by the condenser. To ensure this, operate the instrument according to the following rule of thumb:

Cooling water: max. 20°C **Vapour: 40°C** **Bath: 60°C**

How are these conditions achieved?

1. Set the bath temperature to 60°C.
2. Set the cooling water temperature not higher than 20°C.
3. Allow cooling water to flow through the condenser at approximately 40–50 l/h.
4. Define the operating vacuum in such a way, that the boiling point of the solvent is 40°C.
5. The corresponding pressure can be seen from the Solvent Table in Annexure 1.

Advantages associated with bath temperatures of 60 °C:

a) The evaporating flask can be replaced without risk of burns.
b) The evaporation rate of the water from the heating bath is low (low energy loss).
c) The heating bath energy is used at a good degree of efficiency.

This rule can also be applied to lower bath temperatures, e.g.:

Cooling water: 0°C **Vapour: 20°C** **Bath: 40°C**

HOW TO DISTILL?

Starting

1. To start operating the instrument, the following conditions have to be fulfilled:
   a) All electrical connections are established correctly.
   b) All seals are inserted correctly.
   c) All joints are greased.
2. To start operating the instrument, proceed as follows:
   a) Switch on the instrument.
   b) Set the heating bath temperature to the desired value as described above and wait, until the heating medium has reached its operating temperature. Determine the bath and boiling temperature (consult Solvent Table in Annexure 1).
   c) Allow the cooling water with a temperature not higher than 20°C to flow through the condenser at approximately 40–50 l/h. Turn on the water to the condenser slowly and ensure that it is draining into the appropriate sink. After a couple of minutes the condenser should be cool to the touch.
3. Carry out a vacuum leak test on the apparatus without solvent.
4. Aspirate or fill in solvent you want to distil into the evaporating flask and make sure it does not exceed the filling weight of 3 kg.
5. Set the distillation pressure.
6. Mount the evaporating flask.
7. Set the rotation. Slowly start the rotation and gradually increase the speed.
8. Once the sample has stabilized. Lower the unit so that the sample evaporating flask is exposed to the water. Do not fully submerge the flask. Test the set up by lowering the flask into the water bath. Ensure that the sample flask can freely rotate in the water bath and that the bump trap (if using one) is not rubbing on the side of the water bath. If the system angle needs to be adjusted.
   a. Look on the side of the tower close to the condenser near the back of the rotary evaporator.
   b. Locate the two knobs. The grey one will adjust the angle of the condenser-bump trap-sample flask system. Before continuing follow these steps;
      i. Hold the condenser with your left hand cradling it from the bottom (once the knob is unlocked the condenser will fall in a downwards motion).
      ii. Turn the grey knob with your right hand in a counter clockwise direction (this will unlock the system and allow you to adjust it).
      iii. Gently adjust the angle with your left hand still cradling the condenser from the bottom and your right on the opposite end of the system (near the bump trap).
      iv. Once you are satisfied with the angle hold it as best you can with your left hand still cradling the condenser.
      v. With your right hand lock the knob by turning it in a clockwise direction. Turn only to a finger tight setting.
9. Slowly close the vacuum valve at the top of the condenser on.
10. Watch for bubbles in your sample. Adjust the vacuum as needed. If the distillation product is inclined to foam, lower the vacuum in in steps.
11. Distill

HOW TO DISTILL?

Stopping

1. When the solvent has been completely stripped from the sample slowly open the vacuum valve (up) and continue to run the water for at least 5 minutes.
2. Stop the rotation.
3. Raise the evaporating flask out of the bath.
4. Disconnect the receiver flask from the system and dispose of the solvent into the appropriate waste container.
5. Aerate the system.
6. Switch off the heating bath.

Optimizing the distillation conditions

Depending on the solvent being distilled the distillation might have to be re-optimized.

The condenser should be steamed up to between 2/3 to 3/4. If this is not the case, there are two possibilities to optimize the distillation:

a) When the heating bath has reached 60°C slowly reduce the pressure. Thus, the boiling point of the solvent is reduced and Δ T1 increases resulting in an increase of distillation capacity.
b) When the heating bath has reached 60°C increase the bath temperature. Thus Δ T1 increases resulting in an increase of distillation capacity as well.

When the distillation "dies out"

1. When the distillation "dies out", replace the receiving flask to eliminate the risk of back evaporation. Then, continue distillation.
2. Repeat this process until all desired solvent is distilled off.
3. At the end of the distillation, stop the rotation, pull the flask off and aerate the system.
4. If you do not intend to immediately perform another distillation, turn off the heating bath and cooling water supply to save energy and resources.

HIGH VACUUM & COLD TRAPS

To turn on

1. Check that the cold trap is clean and dry before attaching to the vacuum line.
2. With the Dewar lowered and the system closed to air, turn the vacuum pump on and ensure vacuum performance is leak free (check gauge display).
3. Half-fill the Dewar with liquid nitrogen and carefully raise into position. Never immerse a cold trap in liquid nitrogen unless under vacuum as liquid oxygen (blue in colour) may condense. The result of this can be a violent explosion caused by re-vaporization or by oxidation of organic solvents etc.
4. Attach the apparatus to the manifold via thick walled rubber tubing. Evacuate the apparatus.

To clean trap or shut system down

1. Carefully lower the Dewar and turn the pump off.
2. Vent the system to air and wait for any frozen liquid, which may prevent easy removal of the trap, to thaw.
3. Once the cold trap can be removed, put on a face shield and carefully place the cold trap into an empty Dewar holder to help contain any possible explosion. (Remember that liquid oxygen can be shock sensitive).
4. Carefully transport the cold trap in the Dewar to a fume cupboard and allow to stand overnight behind a safety screen.
5. If the cold trap is likely to have condensed toxic or corrosive vapours, the entire assembly must be placed in the fume cupboard behind a safety screen.
6. Replace with a second clean, dry trap and continue.

Under no circumstances should the system be left open to air with the Dewar containing liquid nitrogen in place.

If liquid oxygen is suspected to have been condensed

1. Notify all laboratory personnel of possible danger.
2. Wear a face shield, fastened laboratory coat and leather gloves.
3. Make sure that the cold trap is no longer in a sealed system. Verify that its contents are either open to the atmosphere or connected to the atmosphere by a pressure relief valve.
4. **Carefully place an empty Dewar box and safety screen around the trap in situ. Do not attempt to remove.**
5. **Leave cold trap to stand overnight vented to air and then carefully re-inspect to check that all suspect material has evaporated**.

MAINTENANCE

Prior to all maintenance work on the instrument switch off the power supply and remove all sources of flammable vapour.

Functional test

Vacuum tightness test

The vacuum tightness test can only be carried out with a vacuum controller installed or when you have a pressure measuring device (manometer) connected to the tube between the pump and the Rotavapor. For this purpose, proceed as follows:

1. Start the instrument and adjust the rotation speed as desired.
2. Apply a vacuum. The instrument is now evacuated while the flask is rotating.
3. Now interrupt the vacuum to the Rotavapor by carefully bending the tube. You should see on the vacuum controller or on the measuring device whether the vacuum in the system remains constant, i.e. the pressure increase per minute should be less than 3 mbar.
4. If the vacuum does not remain constant, check all tube clips, retighten them and grease all ball joints at the condenser side.
5. If that still does not help, replace the seals.
6. Afterwards, repeat the steps described above until the tightness test is passed.

Rotation speed test

To carry out the rotation speed test proceed as follows:

1. Slowly turn the adjusting knob for the rotation speed on the Rotavapor clockwise from the minimum to the maximum setting.
2. At rotations per minute > 20 the motor should turn smoothly at each knob position.
3. When the knob is not turned, the indication of the rotation speed should only change by two digits up or down.
4. In case the rotation speed does not remain constant or there are problems with the motor, call the BÜCHI service providers.

Heating Bath

1. In the oil bath mode, always operate the bath with oil as the heating medium as water might start boiling and evaporating which would lead to heating bath damage.
2. After the oil bath has been standing opened for a prolonged period, condensation water can accumulate at the bottom.
3. When the bath is used again, it must be heated above 100°C with rotating flask to drive the water out.
4. Check the housing for defects (controls, plugs) and clean it regularly with a moist cloth.
5. Never use solvents as cleaning agents as these might damage the instrument.

- Visually examine the tube connections regularly. When tubes become cracked and brittle, replace them with new tubes.
- Grease all joints on the condenser side regularly to achieve optimum tightness of the system.

MAINTENANCE

- When removing and reinstalling the seals, make sure not to damage them. Always move them perpendicularly to the axis of the glass parts and ensure no damage occurs to the sealing lip.
- Never apply grease to the seals and never touch them with sharp object, otherwise they will get damaged.

Cleaning the seals

- To prolong the lifetime of the seals, rinse them regularly with water, especially if “bumping” occurred during the distillation or if working with crystalline products.
- Afterwards, dry them with a soft cloth.

Replacing the seals

- After 3 - 12 months, depending on the wear, the seals should be replaced.
- Seals are subject to wear and tear, thus you should check them regularly and replace them, if necessary, e.g. if they do not pass the vacuum tightness test.

Heating bath

The inner surface of the heating bath should be cleaned if:

- The water bath is calcified or contaminated.
- The oil in the oil bath has changed (colour, viscosity, etc.).
- Light rust spots occur.

1. For this purpose, remove the heating bath from the Rotavapor and empty it.
2. In the case of minor calcifications, use a non-abrasive cleaning agent (e.g. a bathroom cleaner).
3. If the calcification is persistent, use e.g. acetic acid to remove it.
4. Rinse the bath thoroughly afterwards.
5. Rust spots can easily be removed with Scotch-Brite.
6. Make sure to add Borax when using deionized water.

Glass components

To prolong the lifetime of the glass components, consider the following:

1. Clean the seal and glass component regularly, especially after bumping and working with crystalline products.
2. Rinse glass components with water and commercial cleaning agent (e.g. a mild soap solution).
3. Cleaning all the glass components manually.
4. Use an alkaline cleaner to remove dirt, e.g. algae, adhering within the condenser coil.
5. When a thin copper wire is introduced into the condenser coil, the risk of dirt adhering to the condenser coil is reduced.
6. Remove grease from joints. After you have cleaned and completely dried each glass component, visually inspect the components for glass splinters or cracks. Since these components are under vacuum when the Rotavapor is operating, they are subject to strain.
7. Regularly check the glass components for damages and only use glassware that is in perfect condition. Glassware with cracks, stars or other damages can break during operation.

HEALTH & SAFETY

1. Always wear the correct PPE when conducting evaporation.
2. Know where the emergency equipment, fire fighting & chemical spill kit is located.
3. Check the glassware for damages prior to each operation and use only glassware in perfect condition. Glassware with cracks, stars or other damages can break during operation as work under vacuum can result in an implosion with the possible hazard of flying glass, chemical spills and or fire.
4. As soon as the power plug is connected and the mains switch is turned on, the bath starts heating if the actual temperature is below the set temperature. For this reason, make sure that there is always heating medium in the bath to prevent heating bath damage.
5. The heating bath can reach temperatures up to 180°C. To avoid burns, consider the following:
   a) Never remove a rotating flask from the bath because splashing oil can result in burns.
   b) Make sure that no liquid can overflow from the bath when the evaporating flask is submerged.
   c) Install the protective shield (optional accessory) only to a cold heating bath.
19. A setting up to 180°C is only possible in oil bath mode. When using oil baths, do not overheat the oil.
20. Never heat a closed vessel.
21. **DO NOT** distill to dryness or "superheating" of the flask will occur, either cracking the glass or leaving a "tarry" residue which may be very flammable or even explosive.
6. Ensure that all standard taper joints are well greased and clamped into place. The receiver flask should not easily move when under vacuum.
7. Potentially reactive or explosive solvents should be distilled behind transparent explosion shields
8. When possible, distillations involving greater than 500 ml of organic solvents should be carried out in a fume hood.
9. Equipment must be used in the presence of others and not be left unattended.
10. Do not change the immersion angle while the instrument is operating.
11. When the anchoring is loosened the glass assembly can tilt to the left, so that glass breakage can occur. Always support the glass assembly with one hand when you loosen the anchoring.
12. Risk of overflowing: When you are operating with a 5L heating bath, make sure that the controller is configured in a way that the flask is not automatically submerged into the heating bath to avoid an overflowing of the heating bath due to displacement. Introduce the flask manually instead.
13. When a 4L evaporating flask is used the stop-position extension must also be used additionally.
14. Choose the pressure in such a way that the boiling point of the solvent is 40°C (see solvent table).
15. After the set vacuum has been reached, wait for about 1–2 minutes to see whether distillation begins.
16. If the distillation does not start, optimize the parameters (decrease the pressure gradually or increase the bath temperature). Both possibilities lead to an increased distillation capacity.
17. Only use heavy wall Tygon/rubber tubing for vacuum connections.
18. Thin wall Tygon/rubber tubing to be used for all water connections.

HEALTH & SAFETY

19. Flasks of volumes 1L or larger and cooling condenser should be wrapped in tape or a poly coating to restrain glass fragments in case of implosion.
20. For high vacuum systems be sure to add an open/close valve or stopcock between the cold trap and the vacuum pump.
21. Dewars / cold traps are to be filled with a dry ice/ethanol slurry or ice water to condense the solvent. Liquid nitrogen is not recommended as it will liquefy oxygen and freeze most common solvents which will bring about a risk of explosion of the trap.
    a) At atmospheric pressure the temperature of dry ice is -78.5^{0}C.
    b) An ethanol/dry ice slurry has a temperature greater than -80^{0}C.
    c) At atmospheric pressure the temperature of liquid nitrogen is -196^{0}C.
22. When dispensing dry ice exercise extreme care and transfer via provided gloves/mitts.
23. Both the rotation speed and vacuum application should be done gradually when using the rotary evaporator.
24. All the tubing should be in place to both the water and vacuum on the rotary evaporator. Visually inspect the tubing for any cracks and or defects.
25. Ensure that the round bottom flask is at least twice the volume as your sample volume.
26. If need to fill or top up the water bath with deionized water taking care not to fill past the second etch mark on the inner wall of the bath.
27. For low vacuum make sure that the vacuum valve at the top of the condenser is in the ‘open’ position.
28. Excess pressure build up due to too rapid heating and unsafe use of flammable solvents, may result in fires.
29. Very low boiling or more toxic compounds should be distilled only in a fume hood.
30. Fumes leaking through loose joints could come into contact with the heat source and cause a fire.
31. Dropping cold boiling chips through a condenser into hot solutions will result in very rapid boiling and has been known to cause boil over of liquid through the top of the condenser.
19. When finished remove all dangerous substances from the instrument and clean it thoroughly.
20. Store and transport the instrument in its original packaging.
21. Electrical hazard: Always remove the plug connector at the socket first to avoid having energized cables lying about.
22. Dispose of waste correctly.
23. Under no circumstances should the cold trap system be left open to air with the Dewar containing liquid nitrogen in place.

ANNEXURE 1: SOLVENT TABLE FOR 40^{0}C

Solvent	Formula	Molar Mass g/mol	Evaporation Energy J/g	Boiling Point @ 1013mbar	Density g/cm^3	Vacuum mbar for boiling point at 40^{o}C
Acetone	C_3H_6O	58.1	553	56	0.790	556
n-amyl alcohol, n-pentanol	C_5H_2O	88.1	595	37	0.814	11
Benzene	C_6H_6	78.1	548	80	0.877	236
n-butanol	$C_4H_{10}O$	74.1	620	118	0.810	25
2-methyl-2-propanol, tert-butanol	$C_4H_{10}O$	74.1	590	82	0.789	130
Chlorobenzene	C_6H_5Cl	112.6	377	132	1.106	36
Chloroform	$CHCl_3$	119.4	264	62	1.483	474
Cyclohexane	C_6H_{12}	84.0	389	81	0.779	235
Diethyl ether	$C_4H_{10}O$	74.0	389	35	0.714	Atmospheric
1,2-dichloroethane	$C_2H_4Cl_2$	99.0	335	84	1.235	210
1,2-dichloroethylene (cis)	$C_2H_2Cl_2$	97.0	322	60	1.284	479
1,2-dichloroethylene (trans)	$C_2H_2Cl_2$	97.0	314	48	1.257	751
Diisopropyl ether	$C_6H_{14}O$	102.0	318	68	0.724	375
Dioxane	$C_4H_8O_2$	88.1	406	101	1.034	107
Dimethyl formamide, DMF	C_3H_7NO	73.1		153	0.949	11
Acetic acid	$C_2H_4O_2$	60.0	695	118	1.049	44
Ethanol	C_2H_6O	46.0	879	79	0.789	175
Ethylacetate	$C_4H_8O_2$	88.1	394	77	0.900	240
Heptane	C_7H_{16}	100.2	373	98	0.684	120
Hexane	C_6H_{14}	86.2	368	69	0.660	335
Isopropylalchohol	C_3H_8O	60.1	699	82	0.786	137
Isoamylalcohol (3-methyl-1-butanol)	$C_5H_{12}O$	88.1	595	129	0.809	14
Methylethylketone	C_4H_8O	72.1	473	80	0.805	243
Methanol	CH_4O	32.0	1227	65	0.791	337
Methylene chloride, DCM	CH_2Cl_2	84.9	373	40	1.327	Atmospheric
Pentane	C_5H_{12}	72.1	381	36	6.26	Atmospheric
n-propyl alcohol	C_3H_8O	60.1	787	97	0.804	67
Pentachloroethane	C_2HCl_5	202.3	201	162	1.680	13
1,1,2,2-tetrachloroethane	$C_2H_2Cl_4$	167.9	247	146	1.595	35
1,1,1-trichloroethane	$C_2H_3Cl_3$	133.4	251	74	1.339	300
Tetrachloroethylene	C_2Cl_4	165.8	234	121	1.623	53
Tetrahydrofurane, THF	C_4H_8O	72.1		67	0.889	357
Toluene	C_7H_8	92.2	427	111	0.867	77
Trichloroethylene	C_2HCl_3	131.3	264	87	1.464	183
Water	H_2O	18.0	2261	100	1.000	72
Xylene (mixture)	C_8H_{10}	106.2	389			25