

Instructions for the usage of Biotage V10 Evaporator

V10 is a solvent evaporator system enabling evaporation of single samples or pooled volumes.

To date we have 3 types of vials, shown on the top of the instrument, which you can use:

1. Total volume 20 ml – maximal filling volume 8 ml.
2. Total volume 4 ml – maximal filling volume 2 ml.
It cannot be used with the carousel.
3. Custom A (30 ml) – maximal filling volume 12 ml.



Do not use any other type of vials. Do not use volumes above the maximal filling volume to avoid losing your sample and contaminating the system! Do not evaporate larger amounts than a few drops of trifluoroacetic acid!

Start-up

1. Power on the system by the on/off button on the left side of the instrument.
2. When 'Initiate Carousel' is shown on the display press 'Enter' and the carousel will turn around once.
3. Check whether there is an Erlenmeyer flask of fitting size below the condenser to collect solvent rests. Press 'Enter' and the condenser will be emptied into the flask.
4. It takes now 4.5 min for the condenser to cool down; the time is shown on the display.

There are 3 ways to evaporate solvent with this system:

- **A. Mode 0:** Manual load with one prefilled vial (no carousel).
- **B. Mode 0:** Manual lode with a series of prefilled vials using the carousel.
- **C. Mode 1:** Multiple injection from a single sample using the liquid handler.
- **D. Mode 0:** Manual load of samples with very high boiling points (> 160 °C) using the external pump and with one or several prefilled vials (with or without carousel).

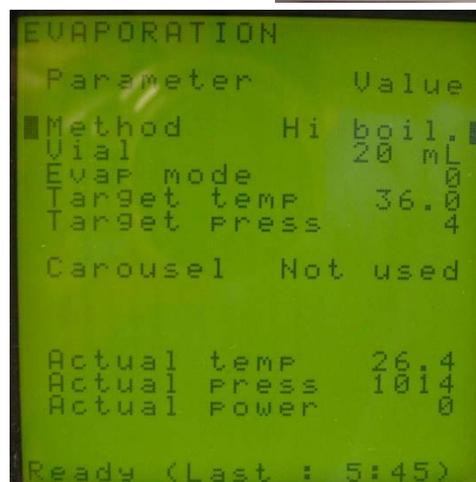
A. Manual load from one prefilled vial (no carousel): "Evaporation mode 0"

1. Ensure that the pump mode 'PERI' is selected (yellow switch).
2. Under **Main Menu** select **Evaporate**, press "Enter", select **Method**,



press "Enter", using the arrows ↓ and ↑ select the method, which fits for your sample. (**Boiling point ranges:** *Very hi boil* > 160 °C; *Hi boil*: 110-160 °C; *Aqueous*: 90-110 °; *Volatile*: 55-90 °C; *Hi Volatile*: <55 °C. For solvent mixtures: *MxdVol* & *HBP*: 55-160 °C; *HPLC fr*: 55-110 °C; *Mixed volatile*: 55-90 °C). Table 1 at the end of this document may provide you further help with choosing a method.

3. Choose **Vial** type. The standard is 20 mL allowing the evaporation of up to 8 mL solvent.
4. Choose **Evap Mode** ("Enter") and select "0" (use ↓ and ↑ for selection, then press "Enter").



- Normally you can go with the target temperature/pressure (**Target temp**, **Target Press**) preselected by the instrument. Would you like to change the target temperature value – for example you have a temperature sensitive compound – select the row **Target temp**, press “**Enter**”, change the value by Pressing ↓ and ↑ for selection, then Press “**Enter**”. To change the **Target Press**, select this row, Press “**Enter**”, change the value by pressing ↓ and ↑ for selection, then press “**Enter**”.



- Select “**Not used**” for the **Carousel** (select this row, Press “**Enter**”, change the value by pressing ↓ and ↑ for selection, then press “**Enter**”).
- Put your vial on the ‘vial seat’. In case you use a vial with lower than 20 ml volume don’t forget the adapters, which you can find in the box on the top of the instrument.
- Press **Start**.
- When you run is finished the text ‘Ready’ is shown on the display. Press ‘**Exit**’ to get back to **Main Menu**.
- Empty the waste container (Erlenmeyer flask) below the drain valve.
- Take your sample tube. In case you used the vial adapters, put them back into the box on the top of the instrument.



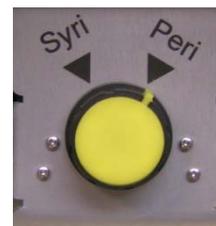
B. Manual mode with a series of prefilled vials using the carousel: “Evaporation mode 0”

- Ensure that the pump mode ‘**PERI**’ is selected (yellow switch).
- Under **Main Menu** select **Evaporate**, press “**Enter**”, select **Method**, press “**Enter**”, using the arrows ↓ and ↑ select the method, which fits for your sample. (**Boiling point ranges**: *Very hi boil* > 160 °C; *Hi boil*: 110-160 °C; *Aqueous*: 90-110 °; *Volatile*: 55-90 °C; *Hi Volatile*: <55 °C. For solvent mixtures: *MxdVol & HBP*: 55-160 °C; *HPLC fr*: 55-110 °C; *Mixed volatile*: 55-90 °C). Table 1 at the end of this document may further help you with choosing a method.
- Choose **Vial** type. The standard is 20 mL allowing the evaporation of up to 8 mL solvent.
- Choose **Evap Mode** (“**Enter**”) and select “0” (use ↓ and ↑ for selection, then press “**Enter**”).
- Normally you can go with the target temperature/pressure (**Target temp**, **Target Press**) preselected by the instrument. Would you like to change the target temperature value – for example you have a temperature sensitive compound – select the row **Target temp**, press “**Enter**”, change the value by pressing ↓ and ↑ for selection, then press “**Enter**”. To change the **Target Press**, select this row, press “**Enter**”, change the value by pressing ↓ and ↑ for selection, then press “**Enter**”.
- Select “**all vials**” for the **Carousel** for up to 16 vials and automatic feeding (select this row, press “**Enter**”, change the value by Pressing ↓ and ↑ for selection, then press “**Enter**”) or “**continual**” if you will keep on feeding the instruments with new vials, by removing the evaporated ones and replacing them with new samples.
- Put your vials on the carousel. Only vials of diameter 28 mm can be used in this mode!
- Press ‘**Start**’.
- When you run is finished the text ‘Ready’ is shown on the display. Press ‘**Exit**’ to get back to **Main Menu**.
- Empty the waste container below the drain valve.
- Take your sample tubes.



C. Multiple injection from a single sample using the liquid handler: "Evaporation mode 1"

1. Ensure that the pump mode '**PERI**' is selected (yellow switch).
2. Check whether there is washing solution ACN:H₂O (95:5)
3. Check whether the waste container has at least 15% free volume. If not empty the content into the solvent waste and place back the empty waste container.
4. Prime the system using **Main Menu - Prime** - press "**Enter**", when the Prime screen is opened press '**Start**'.
5. When the prime operation is completed the text "**Primed**" is displayed at the bottom of the screen. If necessary perform additional primings.
6. Press "**Exit**" to return to the **Main Menu**.
7. Under **Main Menu - Evaporate** select **Method**, Press "**Enter**" and select the method, which fits the boiling point of your sample. Press "**Enter**".
8. Choose **Vial Type** (Press "**Enter**").
9. Set **Evap mode** (evaporation mode) to "1". (select with "**Enter**", confirm with "**Enter**").
10. Normally you can use the target temperature/Pressure (**Target temp**, **Target Press**) preselected by the instrument. Would you like to change the target temperature value – for example you have a temperature sensitive compound – select the row **Target temp**, press "**Enter**", change the value by pressing **↓** and **↑** for selection, then press "**Enter**". To change the **Target press**, select this row, press "**Enter**", change the value by pressing **↓** / **↑**, then press "**Enter**".
11. Choose the amount of solvent you want to evaporate under the option **Total Vol** (total volume of the solvent to be evaporated). Select the desired volume and press "**Enter**". If the total volume is larger than the "**Disp vol**" value, the sample will be evaporated in aliquots. The option "**Auto**" will automatically observe when there is no more solution in your flask. The minimal required amount of solvent system is 10 ml. The solvent should be miscible with the system solvent (CH₃CN:H₂O 95:5).
12. Put the 'needle' on the left of the instrument into your flask.
13. Put your vial on the 'vial seat'. In case you use a vial with lower than 20 ml volume don't forget the adapters, which you can find in the box on the top of the instrument.
14. Press **Start**.
15. As soon as the evaporation finished the text "**Ready**" is shown on the display. Press '**Exit**' to return to the **Main Menu**.
16. Empty the waste container below the drain valve.
17. Take your sample tube.



EVAPORATION	
Parameter	Value
Method	Very Hi
Vial	20 mL
Evap mode	1
Target temp	46.0
Target press	0
Total vol	50.0



D. Evaporation of solvents with very high boiling point (> 160 °C) using the external pump.

The use of the external pump is required for solvents such as 1,2-dichlorobenzene (179 °C), 1-methyl-2-pyrrolidinone (NMP, 202°C), and dimethylsulfoxide (DMSO, 189 °C).

1. Ensure that the yellow switch is pointing on 'PERI'.
2. Select **Main Menu – Configuration – Automation – External vacuum pump** and change from "No" to "Yes".
3. Return to the **Main Menu** pressing the 'Exit' four times.
4. Under **Main Menu** select **Evaporate** ("Enter") and then select **Method**, press "Enter" and using the arrows ↓ and ↑ select the method 'Very hi boil'.
5. Choose **Vial** type. The standard is 20 mL allowing the evaporation of up to 8 mL solvent.
6. Choose **Evap Mode** ("Enter") and select "0" (use ↓ and ↑ for selection, then press "Enter").
7. Normally you can go with the target temperature/Pressure (**Target temp**, **Target press**) preselected by the instrument. Would you like to change the target temperature value – for example you have a temperature sensitive compound – select the row **Target temp**, press "Enter", change the value by pressing ↓ and ↑ for selection, then press "Enter". To change the **Target Press**, select this row, press "Enter", change the value by pressing ↓ and ↑ for selection, then press "Enter".
8. Select "Not used" for the **Carousel** (select this row, press "Enter", change the value by pressing ↓ and ↑ for selection, then press "Enter"). Alternatively you can use several samples and the carousel as explained above (B.6.).
9. Start the pump below the table.
10. Put your vial on the "vial seat". In case you use a vial with lower than 20 ml volume don't forget the adapters, which you can find in the box on the top of the instrument.
11. Press **Start**.
12. When your run is finished the text 'Ready' is shown on the display press 'Exit' to get back to **Main Menu**.
13. Turn off the pump below the table.
14. Select **Main Menu – Configuration – Automation – External vacuum pump** and change from "Yes" to "No".
15. Empty the waste container below the drain valve.
16. Take your sample tube.
17. In case you used the vial adapters put them back into the box on the top of the instrument.



F. Some useful tips

1. The "pause button"  will pause an ongoing job following the finish of the ongoing evaporation. Pressing the "play button"  will restart the job.
2. The "stop button"  will stop the run immediately and the sample vial will return to its starting position.
3. To clean the tubes of the system use **Main Menu – Clean**.
4. Following the evaporation of a volatile solvent it may be difficult to get down to zero pressure. To fix the vacuum start an evaporation cycle using an empty vial.
5. The 'Exit' button will take you one step back in the menu.

Turn off the system for weekends and for longer holidays as Christmas or Eastern.

Comparison with other types of evaporation:

Solvent	BP (° C)	Biotage V-10 evaporation	GeneVac evaporation	Blow-down evaporation
NMP	202	18 minutes	N/A	N/A
DMSO	180	15 minutes	180 minutes	N/A
DMF	150	4 minutes	90 minutes	N/A
Pyridine	115	5.5 minutes	70 minutes	N/A
Water	100	9 minutes	140 minutes	240 minutes
Methanol	65	3 minutes	70 minutes	40 minutes
Cyclohexane	81	2.5 minutes	40 minutes	20 minutes

Bilaga 1.

Tabell 1. Guide för val av metod beroende på kokpunkt hos lösningsmedel.

Lösningsmedel	Kokpunkt (°C)	Metod	Kräver extern vakumpump
1,2-Dichlorobenzene	179	Very hi boil	Ja
1,2-Dichloroethane DCE	83.5	Volatile	
1,4-Dioxane 1,4-diox	101.5	Aqueous	
1-Methyl-2-pyrrolidinone NMP	202	Very hi boil	Ja
Acetic acid HOAc	117.9	Hi boil	
Acetone Propanone, 2-propanone	56.1	Volatile	
Acetonitrile ACN MeCN	81.6	Volatile	
Ammonia NH ₃	33.3	Hi Volatile	
Butan-1-ol n-BuOH	117.7	Aqueous	
Butan-2-ol	99.5	Aqueous	
Chloroform	61.1	Volatile	
Dichloromethane DCM	40	Hi Volatile	
Diethyl ether	34.5	Hi Volatile	
Dimethyl sulfide	37.3	Hi Volatile	
Dimethyl sulfoxide DMSO	189	Very hi boil	Ja
Ethanol EtOH	78.2	Volatile	
Ethyl acetate EtOAc, AcOEt	77.1	Volatile	
Formic acid HCOOH	101	Aqueous	
Heptane	98.5	Aqueous	
Hexane	68.7	Volatile	
Hydrogen peroxide H ₂ O ₂	150.2	Hi boil	
Isopropanol IPA (isopropyl alcohol),	82.3	Volatile	
Methanol MeOH	64.6	Volatile	
Methyl t-butyl ether MTBE, t-butyl methyl ether	55.2	Hi volatile	
N,N-Dimethylacetamide DMA or DMAc	165	Hi boil	
N,N-Dimethylformamide DMF	153	Hi boil	
Pentane	36.1	Hi volatile	

Propanoic acid Propionic acid	141.2	Hi boil	
Pyridine	115.2	Aqueous	
Tetrahydrofuran THF	65	Volatile	
Toluene PhMe Aqueous	110.6	Volatile	
Trifluoroacetic acid TFA	73	Volatile	
Trimethoxymethane TMM (trimethyl orthoformate) 104.0	104	Aqueous	
Water H2O	100	Aqueous	

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Tabell 2. Tabell över kokpunkt hos azeotroper

Azeotroper med vatten:

Komponent nr.2	Bp blandning
Etanol	78,1°C
Acetonitril	76,5°C
DCM	38,8°C

Azeotroper med etanol:

Komponent nr.2	Bp blandning
Acetonitril	72,9°C

Azeotroper med metanol:

Komponent nr.2	Bp blandning
Acetonitril	63,5°C