



Discover LabMate Installation

Read this guide carefully and thoroughly prior to operating this product. Retain it for future reference. In addition to this information, a complete Discover Operation Manual (CEM part # 600155) is provided with the Discover instrument. CEM also provides technical and applications support for synthesis related questions by a team of trained chemists. For applications support, call (800) 726-3331 (inside the US) or (704) 821-7015 and request "Synthesis Applications," or send an email to synthesis.support@cem.com.

Installation Site

The Discover System can be installed in a laboratory fume hood or on a laboratory bench with proper ventilation. Choose a location that

- provides at least 8 in (20 cm) of open space on each side and 6 in (15 cm) of open space in the rear of the instrument for ventilation,
- is free from vibration of large equipment and/or excessive walk-through traffic,
- provides a temperature range of 41 °F (5 °C) to 104 °F (40 °C) and a humidity range of 10-85% relative humidity,
- provides adequate space for sample handling (and computer placement if applicable), and
- permits the system to be connected to a dedicated, grounded 120 VAC outlet. The Discover System should be operated on a stabilized, constant voltage AC power supply. To operate properly, the voltage must be within $\pm 10\%$ of the specified level.

Tools Required: Razor blade or box cutter.

Unpacking

1. Open the shipping carton containing the Discover LabMate.
2. Remove the packing material.



3. Carefully remove the Discover System from its shipping carton.



4. Place the Discover in a fume hood or on a laboratory bench with proper ventilation.
5. Keep all the packing materials until the system has successfully been installed.

Inspect the instrument for shipping damage such as cracks, dents or warping. If the instrument has been damaged in shipping, contact the freight carrier to report the damage and to file a damage report. Contact CEM to report damage at 800-726-5551

WARNING

If damage to the instrument is noted upon receipt, do not attempt to operate the instrument.

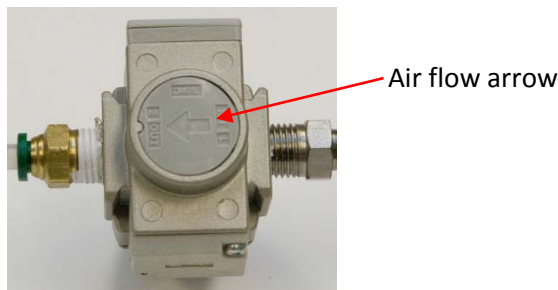
Never modify the attenuator access port or insert metallic objects such as wire into the port. Serious microwave leakage and/or electrical shock may result. The access port of the Discover is electrically grounded to the microwave cavity and is designed to prevent leakage of microwave energy.

Cardiac pacemakers require magnets to control operation during checkout. Because the Discover System is equipped with a variable-speed, electromagnetic sample stirrer, some danger exists if a pacemaker is positioned in close proximity to the instrument cavity. If the instrument is suspected of interfering with the operation of a pacemaker, the instrument should be turned off or the pacemaker wearer should move away from the instrument.

Setup

1. Remove the following items from the accessory kit:
 - Regulator Assembly (541455-M)
 - Fittings for the air line (BR163380, BR862000, BR862005)
 - Tubing (BR221221)
 - Power Cord (116620)
2. Plug the power cord into the back of the instrument.
3. Plug the power cord into the electrical outlet.
4. Set up the air regulator (541455-M) as follows:

NOTE: The regulator has an arrow printed on it to show the direction that the air must flow. Set up the regulator according to the arrow.



- a. Cut a perpendicular 1' section off of the 8' of 1/4" tubing (PN BR221221).

- b. Place the 1' section of tubing into the left side of the Discover by pushing the tubing into the opening until resistance is felt.



- c. Connect the opposite end of the 1' section of tubing to the regulator. The regulator has an arrow printed on it indicating the direction of air flow. Position the regulator with the arrow pointing toward the Discover.
d. Connect the airline to the opposite end of the tubing connected to the regulator.



NOTE: There are 2 regulator setup options. The option on the left illustration above is set up to connect Tygon tubing (BR870002) over a barb fitting (BR802008). A clamp (BR851037) is used to secure the connection to the barb fitting. The regulator setup on the right uses ¼" tubing (BR221221) designed for use with most standard NPT connections.

- e. Connect the opposite end of the tubing to the air source.
f. To change the amount of air flow from the regulator:
1. Pull out the knob on the regulator.
2. Rotate the knob on the regulator clockwise to increase the amount of air flow and counter-clockwise to decrease the amount of air flow. The minimum compressed gas requirement is 25 PSI (20L/min flow) and the maximum pressure is 60 Psi. The air flow should remain on at all times when a reaction is being performed.
3. Push the knob in to set the desired pressure.
5. Remove the piece of brown paper from the center of the cavity.



6. Check the cavity for Debris. Refer to the LabMate manual (CEM part # 600155) for information on removing debris from the cavity.
7. Locate the 9 pin connector extending from the IntelliVent pressure device. Record the "slope" and "intercept."

8. Plug the 9-pin connector extending from the IntelliVent pressure device into the back of the instrument.



9. Using the power switch located on the left side of the Discover, turn the instrument on.
10. Press the EDIT key on the Discover keypad.
11. Press the Right Arrow key to display "Pressure."
12. Use the arrow keys to select "Enter Calibration."
13. Press the ENTER key.
14. Press the EDIT key to highlight the slope calibration data. Using the numeric keypad, enter the value for the slope of the pressure curve.
15. Press the ENTER key.
16. Using the numeric keypad, enter the value for the intercept.
17. Press the ENTER key.
18. For additional control of the Discover system, use the "Synergy Application Software" manual (600177) for connection and operation of the Synergy software.

The system is ready to operate. Perform the "System Verification" to ensure that the instrument is operating correctly.

System Verification

Solvent Testing verifies both the temperature and pressure calibration values. It should be used as a check to ensure both means of measurement are operating correctly. However, it is not necessarily an indication as to which is erroneous.

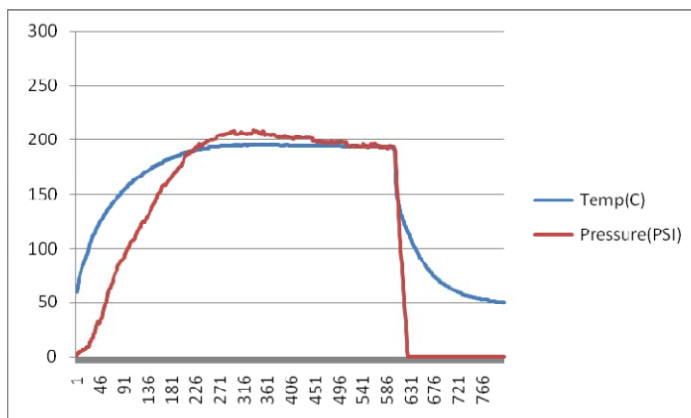
Items Required for Solvent Test:

- Water
- 10mL vial with cap
- Stir bars
- 10mL attenuator

Refer to the section of these instructions titled “Perform a Closed Vessel Reaction” for additional instructions on performing vessel setup.

1. Place 3 ml of water into a 10 ml reaction vessel with a stir bar.
2. Cap the reaction vessel.
3. Using standard (dynamic if using Synergy) method control, program a method with 300 W, 10 minute hold time (10 minute ramp if applicable), 200°C and 250 psi. Stirring should be set on high.
4. Place the 10 ml tube into the Discover cavity and press the START key to begin the method.
5. The sample should heat to 200°C and ~200 psi ($\pm 10\%$).

NOTE: If the sample is not within $\pm 10\%$, contact CEM Corporation (800) 726-3331 (inside the US) or (704) 821-7015.



Perform a Closed Vessel Reaction

- Items Required:
- 10mL Attenuator
- 10mL Vial
- Stir Bar appropriate to vial size
- 10mL Cap

Vessel Preparation

1. Place the stir bar into the vessel
2. Place the reaction components into the vessel
3. Place the cap on the vial

Note: The working volume for the 20mL vial is 0.2mL to 7mL.



WARNING

Proper precautions must be taken to avoid contact with solvents or solvent vapors. Protective gear should be worn as outlined in the user's safety program for hazardous materials and the reagent manufacturer's material safety data sheet. Refer to these guidelines for proper decontamination, handling and disposal of reagents or any hazardous materials.



WARNING

The high temperature spill cup (162426) is required for reactions exceeding 200 C for greater than 20 minutes or programmed temperature greater than 250 C. When replacing the spill cup, the IR sensor must be verified and possibly recalibrated.

The Discover LabMate can be used only with CEM-supplied 10mL reaction vessels and septa designed for high temperature and/or pressure reactions in a microwave.

4. Install the appropriate assembly.



5. Rotate the attenuator assembly clockwise until the attenuator locks into position.

6. Place the vessel into the attenuator.



7. Position the IntelliVent over the vessel.



Microwave Irradiation

1. Load or create the appropriate method into the instrument software of the Discover SP. Refer to the section “Load Method” or “Create New Method” for creating a method.
2. Press the START key. The IntelliVent will close and lock onto the vessel. The “Waiting” screen will appear prior to irradiation initiation. Once the method begins, the system will ramp to the set-point temperature. The system will maintain the programmed parameters to perform the complete method.

NOTE: If desired, during the method, the “hot keys” can be pressed to edit the method parameters (temperature, time, pressure, and/or power).

Vessel Removal

1. Once the vessel is properly cooled the IntelliVent will release the vessel.
 - If the IntelliVent does not automatically release the reaction vessel, the pressure (or temperature) is above the release limit. A message will appear indicating the current temperature and pressure value. Cool the reaction vessel completely, then manually release the IntelliVent.

WARNING

To prevent the possibility of severe burns, ensure that insulated gloves and protective gear as outlined in the user’s safety program are worn.

2. Carefully remove the vessel from the attenuator.

NOTE: If the cap still holds residual pressure, place the cap and vial in a fume hood or other well ventilated area. Then use a needle to pierce the septa and vent.

3. Remove the cap from the vessel by pressing up on one side of the cap.

WARNING

Vessels and caps are designed for only one use. Do not use vessels and/or caps more than one time.

Perform a Closed Vessel Reaction

Items Required:

10mL Attenuator

10mL Vial

Stir Bar appropriate to vial size

10mL Cap

Vessel Preparation

1. Place the stir bar into the vessel
2. Place the reaction components into the vessel
3. Place the cap on the vial

Note: The working volume for the 20mL vial is 0.2mL to 7mL.



WARNING

Proper precautions must be taken to avoid contact with solvents or solvent vapors. Protective gear should be worn as outlined in the user's safety program for hazardous materials and the reagent manufacturer's material safety data sheet. Refer to these guidelines for proper decontamination, handling and disposal of reagents or any hazardous materials.



WARNING

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The Discover LabMate can be used only with CEM-supplied 10mL reaction vessels and septa designed for high temperature and/or pressure reactions in a microwave.

4. Install the appropriate assembly.
5. Rotate the attenuator assembly clockwise until the attenuator locks into position.

- f. Press the HOME key until the Discover Home screen is displayed.

NOTE: To return to use of the IntelliVent Pressure Device, complete the above procedures but select “N” in in step d.

Microwave Irradiation

1. Load or create the appropriate method into the instrument software of the Discover. Refer to the section of this document entitled “Load Method” or “Create New Method” for creating a method.
2. Press the START key. The “Waiting” screen will appear prior to irradiation initiation. Once the method begins, the system will ramp to the set-point temperature. The system will maintain the programmed parameters to perform the complete method.

NOTE: If desired, during the method the “hot keys” can be pressed to edit the method parameters - temperature, time, pressure, power and stirring.

Vessel Removal

1. Upon completion of the reaction, wait until the temperature setpoint has been obtained and the cool-down cycle has terminated.
2. If applicable, remove the condenser.
3. Remove the attenuator by rotating it counterclockwise.
4. The round bottom flask can then be removed from the cavity and the reaction work-up can be performed.

WARNING

To prevent the possibility of severe burns, ensure that insulated gloves and protective gear as outlined in the user's safety program are worn.

5. Remove the Teflon® disk from the cavity.

NOTE: Teflon is a register trademark of the Dupont Corporation.

Microwave Tips

- Cover all solids in reaction vessel with liquid
 - ❖ Metal catalysts can be used, but ensure they are wetted or in solution
- Never exceed the maximum working volume of the vial
- Always use a stir bar that adequately mixes the reaction contents in the microwave
- Reactions can be performed under an inert atmosphere
 - ❖ Purge the vessel prior to performing a reaction
- Try performing the reaction neat or at higher concentrations
- When transitioning a conventional, open vessel reaction to a closed vessel reaction:
 - ❖ Increase reaction temperature 25°C above the highest boiling point solvent in vial
 - ❖ Start with 150 watts and hotkey higher if necessary

When to Exercise Caution

- Anything that would be a concern conventionally, regardless of reaction size
- High concentrations of acids, bases, or salts
 - ❖ Includes other ionic or very polar species
 - ❖ Bases react more readily in a microwave
 - ❖ Generally, 10% or less is recommended
- Gases formed during the reaction
 - ❖ Use an open vessel format to relieve excess unnecessary gases
 - ❖ Increase headspace (empty volume in vessel) to accommodate the generated gas

How to Cautiously Microwave

- Start with a low power input
 - ❖ 50 W or less (this can be adjusted as necessary later)
- Watch the first minute of the reaction
- Use the hotkeys during the reaction
 - ❖ Adjust the power
 - ❖ Lower the temperature and/or pressure limits
 - ❖ Raise the temperature and/or pressure limits
- If a gas forms during the reaction
 - ❖ Cool the reaction completely
 - ❖ Allow the ActiVent to release the excess gas

	Total Volume/ Vessel Name	Working Volume	Sealed/Pressurized or Open/Atmospheric
Standard Vessel	10 mL	0.200 - 7.0 mL	Sealed
Open Vessel	Open Vessel-up to 125 mL Round bottom flask with up to a 24/40 ground glass joint	60% of vessel volume	Open

Microwave Absorbance	Solvent
High (100 Watts)	DMSO, EtOH, MeOH, Propanols, Nitrobenzene, Formic Acid, Ethylene Glycol
Medium (150 Watts)	Water, DMF, NMP, Butanols, Acetonitrile, HMPA, Methyl Ethyl Ketone, Acetone and other ketones, <i>o</i> -Dichlorobenzene, 1,2-Dichloroethane, 2-Methoxyethanol, Acetic Acid, Trifluoroacetic Acid
Low (300 Watts)	Chloroform, Dichloromethane, Carbon Tetrachloride, 1,4-Dioxane, THF, Glyme, and other ethers, Ethyl Acetate, Pyridine, Triethylamine, Toluene, Benzene, Chlorobenzene, Xylenes, Pentane, Hexane and other hydrocarbons

Create a New Method

The Discover System has multiple control options for programming a method; however, the Standard Control option is the recommended control techniques for routine operation.

Standard

The Standard Control option provides more flexibility in how the user programs a reaction method and makes greater use of the feedback control data from the temperature and pressure systems. It applies a specified amount of power, defined by the user, to reach the control point. It modulates this selected power automatically, based on the sensor feedback data, to ensure the control point is reached rapidly, but with limited error (temperature or pressure “overshooting”). The user programs:

- the maximum amount of microwave power that can be applied to the method,
- a temperature control point,
- a pressure control point,
- a run time (maximum amount of time system allows to reach temperature or pressure setpoint)
- a hold time (the time the system maintains the control parameters),
- a stirring function with speed control
- PowerMAX (simultaneous cooling).

The Standard Control option can be programmed for up to five (5) stages for multiple irradiation steps and is a general control method for maintaining critical control points.

1. With the main menu displayed, press the “file” key.
2. Using the left arrow key, toggle and select “New Method” to create a new method.
3. Press the ENTER key.
4. Using the right arrow key, toggle and select “Standard.”
5. Enter the desired method parameters using the ARROW keys to navigate between temperature, time, power, pressure, stirring, PreMix, PowerMAX and number of Stages. Use the “+/-” keys to increase and decrease the numeric value.
 - Power- 0-300W
 - Temperature- 0 - 300 °C
 - Pressure “P”- 0-300PSI or 0-21Bar
 - Run Time- 1 second to 59 minutes and 59 seconds
 - Hold Time- 1 second to 59 minutes and 59 seconds
 - Stirring- “high”, “medium”, “low” and “off”
 - Cooling (PowerMAX)- “on” or “off”
 - Stage “Stage”- 1-5 stages
6. Using the Right arrow key, toggle and select “Y”(yes) or “N” (no) to save the method.
7. Press the ENTER key. If “no” is selected, the instrument main screen will appear with the method title “untitled.” If “yes” is selected, proceed to step 8.
8. Using the arrow keys, highlight the first letter of the method name.
9. Press the ENTER key.
10. Continue to use the arrow key(s) and the ENTER key to complete the name of the method (12 characters maximum).
11. When the name is completed, use the arrow key(s) to highlight “Exit.”
12. Press the ENTER key.
13. Once the method name is selected, press the ‘Home’ key.

Load Method

A method that has been previously saved can be recalled for future reaction.

1. With the Discover home screen displayed, press the “file” key.
2. Using the arrow keys, toggle and select the method to be loaded.
3. Press the ENTER key.
4. The main menu is displayed with the loaded method name in the title bar.