

Manual





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Safety Notations

This manual uses safety alert words at points in the documentation where the user should be aware of potential hazards.

NOTE

A **NOTE** is intended to provide emphasis on procedures that may be misinterpreted or overlooked, or to clarify confusing situations.

CAUTION

A **CAUTION** is intended to provide essential information and to emphasize procedures that, if not strictly followed, may result in improper instrument operation.

A **WARNING** is intended to emphasize dangerous or hazardous conditions that may result in personal injury to the user and/or damage or destruction of the instrument.

A PINCH POINT

A PINCH POINT is intended to keep hands clear during operation and if not avoided, could result injury.

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Surface is hot to the touch.

About the Instrument

The Discover[®] 2.0 is designed to enhance the ability to perform chemical reactions under controlled conditions on a laboratory scale. The system facilitates either homogeneous or heterogeneous solution phase chemistry, or chemistry conducted on solid supports. It accommodates vessels ranging in volume from 5 mL to 125 mL for reactions performed under atmospheric conditions and 10-mL, 35-mL, or 100-mL vessels for reactions performed at elevated temperatures and pressures. The Discover 2.0 incorporates Activent[®] Technology which permits the user to release unwanted gaseous byproducts from the reaction to prevent over pressurization and vessel failure from gaseous buildup. This ensures safe handling of the vessel during and at the end of the reaction. The iWave[®] temperature sensor is a contactless, volume-independent sensor that accurately measures the temperature during the run.

Primary uses of the Discover 2.0 are in the discovery and lead optimization phases of the new product development process. Microwave energy is applied to the vessel contents (reactants, catalysts, salts, solvents and/or solid supports) to accelerate the chemical reaction. The microwave absorption properties of some liquid and solid materials, due to their polar and ionic characteristics, have the capability to significantly enhance chemical reactions relative to traditional energy application (heating) techniques. The microwave interaction properties with the reactants, intermediates, catalysts, solid supports and salts provide unique opportunities for the synthetic chemist.

This manual is intended for use by both novice and experienced users for the operation and maintenance of the Discover 2.0. Additional information, including but not limited to: training notes and videos, application notes, software updates, and parts can be viewed on CEM's website at http://cem.com/Discover-2. This manual contains proprietary information which shall not be reproduced or transferred to other documents or disclosed to others without prior written permission from CEM Corporation.

This manual refers to Discover 2.0 software version 1.01 for all software information, including screenshots and technical information.

The English-language version of this manual is the official version. In the event that this manual is translated into other languages, such translations are not official, and the English-language version is controlling in the case of any ambiguity or inconsistency.

Read and fully understand all documentation before operating the instrument.

Common Abbreviations

Abbreviation	Definition
°C	degrees Celsius
°F	degrees Fahrenheit
А	ampere
cm	cenitmeter
Hz	Hertz
kg	kilogram
m	meter

Abbreviation	Definition
mL	milliliter
mm	millimeter
lb	pounds
psi	pounds per square inch
VAC	volts of alternating current
W	watts

Important Safeguards

General guidelines for safe operation of the Discover 2.0 are presented below and all specific safety messages are located throughout the manual.

	Open Vessel	10-mL	35-mL	100-mL
Minimum Working Volume	2.0 mL	0.2 mL*	2.0 mL	15 mL
Maximum Working Volume	75% of vessel volume	7.0 mL	25.0 mL	70 mL
Maximum Temperature	300 °C	300 °C	300 °C**	250 °C
Maximum Pressure	N/A	435 psi	300 psi	250 psi
Maximum Power	300 watts	300 watts	300 watts	300 watts

*The minimum volume is 0.4 mL unless the optional 4-mL spacer kit is used.

**35-mL liners are limited to 200 °C and 200 psi.

Instrument Safeguards

- Service can only be performed by an authorized CEM service technician. This instrument utilizes high voltages and microwave irradiation.
- \cdot DO NOT tamper with the instrument.
- · If any damage to the instrument is noted, do not attempt instrument operation.
- · Use only CEM specified consumables and accessories.

Sample Preparation Safeguards

- 10-mL and 35-mL vessels and caps are designed for only one use. Do not use these vessels and/or caps more than one time.
- 100-mL vessels may be reused but the caps are single use only and must not be reused.
- Proper precautions must be taken to avoid contact with reagents or reagent vapors. Protective gear should be worn as outlined in the user's safety program for hazardous materials and the reagent manufacturer's safety data sheet. Refer to these guidelines for proper handling and disposal of the reagents. Dispose of all waste in accordance with all applicable local, state, and federal health and safety recommendations.
- 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 vessel.
- 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.
- · Liners are recommended for chemistry incompatible (ie. zeolite synthesis) with the pyrex vessel.
 - Liners must be purchased directly from CEM Corporation or through its authorized dealer network.
 - Liners are only available for the 35-mL vessel and are designed for multiple uses unless visually compromised.

Microwave Heating Safeguards

- 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 to adjust reaction parameters, if necessary.

- · Cool the reaction completely prior to vessel removal.
- \cdot Allow the Activent to release any excess gas.
- Use the "Microwave Method Calculator" located in the software to convert conventional methods to microwave methods.

Selected Solvent Chart

Microwave Absorbency	Solvent			
High	DMSO, EtOH, MeOH, propanols, nitrobenzene, formic acid, ethylene glycol			
Medium	Water, DMF, NMP, butanols, acetonitrile, HMPA, methyl ethyl ketone, acetone and other ketones, o-dicholorbenzene, 1,2-dichloroethane, 2-methoxyethanol, acetic acid, trifluoroacetic acid			
Low	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			

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
 - \cdot Use an open vessel format to relieve excess unnecessary gases
 - · Increase headspace (empty volume in vessel) to accommodate the generated gas

Instrument Overview

Front and Side View



	Item	
1	Touch Screen Display	Shows menus, method parameters, etc.
2	Attenuator Port	Provides access to the instrument cavity while the interlocks prevent microwave emission when the attenuator port is not properly installed
3	Activent	Automated pressure device. The Activent safely relieves the solvent/vapor from the reaction vessel and releases it through the vent tube and into a controlled environment.
4	Power Switch	Turns AC power on and off to the instrument
5	Camera Focus Switch	Allows user to manually clarify the image
6	USB Port	Import, export, backup and/or save instrument data

Back and Side View



	Item	Description
1	Speaker	Audio output to alerts users
2	Ethernet Port	Connects peripheral accessory and allows communication and connection to an external computer for data collection, a local area network (LAN) or the Internet. (Optional)
3	USB Port(s)	Import, export, backup and/or save instrument data
4	Serial Port	Connects peripheral accessory
5	Peripheral Connections	Connects peripheral accessory
6	Cooling Fan	Draws room air past the electronics
7	Spill Tray	Removable collection tray for cavity contents in case of a vessel failure
8	Nameplate	Lists model, serial number, operating voltage, frequency, and current draw
9	Power Cord Receptacle	Receives the female end of the power cord
10	Cooling Gas Line	Provides a hose connection for the cooling gas source
11	Fuses	Prevents power overload for Autosampler
12	Electrical Connector	Provides AC power to the Autosampler
13	Vent Tube	Directs solvent/vapor from the instrument into a designated container

Accessories



10-mL Cap



35-mL and 100-mL Cap



10-mL Vessel



35-mL Vessel



10-mL Attenuator



35-mL Attenuator



100-mL Vessel



Open Vessel Disk



100-mL Attenuator and Collar



Open Vessel Attenuator



100-mL Attenuator Stand

Home Screen

The Home screen is the first screen that appears when the system is turned on. Four (4) areas are accessible from this screen: Methods, Run, Results, and System Menu.



- · Methods: Create, edit or delete specific conditions to be used for a given synthesis
- Run: Allows users to run a previously created method.
- Results: View and export past run data
- System Menu: Provides access to any functionality that does not directly involve method development, running a
 method or viewing method results

Methods



Run



Results



System Menu



- · About: Contains current software version, system information and instrument documentation
- Home: Returns to the Home screen
- Settings: Adjustable system settings necessary prior to running samples, system information including serial number, software version, contact information, etc., and users
- Tools: System Security settings and hardware
- $\boldsymbol{\cdot}$ Users: Location for creating and deleting different user accounts
- Help: Import/Export Data, create troubleshooting bundles and view system data
- · Log out: Protects the current user's access or prevent unauthorized actions on the current login session
- Exit: Shuts down system or restarts system/software

Unpacking

- **1.** Select a location that meets requirements as outlined in the Site Preparation Guide (P/N FM0072) or Installation Guide (P/N 601008) before installing the instrument.
- **2.** While unpacking, inspect the box, accessory kit and instrument for damage that may have occurred during shipping. If you notice damage, DO NOT attempt instrument set up. Contact your CEM representative for help.
- 3. Remove the accessory kit from the box using the handhold cutouts. Lift straight up to remove.
- 4. Remove the box containing the power cord and sit it aside.
- **5.** Place one hand under the instrument in the front and the other under the instrument in the back. Lift the instrument straight up while a second person holds the shipping box on the side of the box.
- **6.** The packing foam will come up with the system. Once the instrument is out of the box, remove the foam from the sides.
- 7. Place the instrument on the bench top and remove the plastic sheeting from the instrument.
- 8. Plug the power cord into the back of the instrument and into a dedicated, grounded outlet.
- **9.** Position the instrument facing forward. Make sure the power switch can easily be accessed. Turn the power switch to ON.
- **10.** Wait for the instrument to start up. Follow the instructions in the instrument software to finalize the installation.

NOTE

The Discover 2.0 Manual is located in the "Help" section of the software.

System Setup

- 1. Install the vent tubing.
 - **1.1.** Remove the nut and ferrule from end of the white connector that is not connected to the tubing.
 - **1.2.** Locate the vent tubing extending from the back of the instrument.
 - **1.3.** Place the nut and ferrule on the tubing extending from the back of the instrument. Ensure that the orientation of the nut and ferrule matches.
 - **1.4.** Connect the tubing extending from the back of the instrument to the vent line.



- **1.5.** Place the opposite end of the tubing in the back of the fume hood or other location appropriate for solvent exposure.
- **2.** Install air regulator.

- **2.1.** Locate the 8' of ¹/₄" tubing (P/N BR221221).
- **2.2.** Cut a perpendicular 1' section from the tubing.
- **2.3.** Place the 1' section of tubing into the left side of the Discover 2.0 by pushing the tubing inward until resistance is felt.
- **2.4.** Connect the opposite end of the 1' tubing to the regulator. The regulator has an arrow printed on it to indicate the direction that the air must flow. Position the regulator with the arrow pointing toward the instrument.
- **2.5.** Connect the tubing to the opposite end of the regulator. There are 2 regulator setup options.
 - Connect the Tygon tubing (P/N BR870002) over a barb fitting (P/N BR802008) then use a clamp (P/N BR851037) to secure the connection to the barb fitting.
 - Connect the ¹/₄" tubing (P/N BR221221) designed for use with most standard NPT connections.
- **2.6.** Connect the opposite end of the tubing to the air source.
- **2.7.** Adjust the amount of air flow from the regulator.
 - **2.7.1.** Pull the knob out.
 - 2.7.2. 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 (20 L/min flow) and the maximum pressure is 60 psi. The air flow should remain on at all times when a reaction is being performed.
 - **2.7.3.** Push the knob back in to set the desired pressure.
- **3.** Remove the attenuator from the cavity by rotating it counterclockwise and then lift straight up.



4. Remove the thumb screw from the bottom of the attenuator.



5. Remove the 35-mL shipping device and place the screw back into the shipping device. Retain the 35-mL shipping device for future use.



- **6.** Check the cavity for debris.
- **7.** Adjust the camera focus.
 - **7.1.** Verify that the camera is enabled. Select Settings -> Accessories -> Camera. Toggle the "Camera Window" to ON.
 - **7.2.** Select the floating camera button located in the Discover 2.0 software.
 - **7.3.** Manually adjust the camera focus using the camera focus switch on the left side of the instrument.
- **8.** The Discover 2.0 is now ready for use.

Proper precautions must be taken to avoid contact with reagents or reagent vapors. Protective gear should be worn as outlined in the user's safety program for hazardous materials and the reagent manufacturer's safety data sheet. Refer to these guidelines for proper handling and disposal of the reagents. Dispose of all waste in accordance with all applicable local, state, and federal health and safety recommendations.

Reaction Preparation

- **1.** Select the vessel size: 10-mL, 35-mL, 100-mL, or open vessel. 10-mL and 35-mL vessels are designed for only one use. Do not use vessels more than one time.
 - 10-mL vessel: 0.2 mL 7 mL
 - · 35-mL vessel: 2 mL 25 mL
 - · 100-mL vessel: 15 mL 70 mL
 - · Open Vessel up to 125 mL: 2 mL 75% of vessel volume
- **2.** Place an appropriately sized stir bar into vessel. Always use a stir bar that adequately mixes the reaction contents in the microwave.
- **3.** Add the reagents, reactants, and solvent to the vessel. Metal catalysts can be used, but ensure they are wetted or in solution. Ensure that all solids have been washed from the surface of the vessel prior to beginning the reaction.



Prepare the Vessel and Microwave Cavity

10-mL and 35-mL Closed Vessel Reactions

Reactions can be performed under an inert atmosphere. If desired, prepare the vessel in a glovebox or pierce the cap and purge the vessel prior to performing a reaction.

- **1.** For 10-mL and 35-mL closed vessel reactions only, place the cap onto the vessel.
- 2. Place the vessel into the attenuator.

CAUTION

Use only the 35-mL attenuator with the 35-mL vessel. DO NOT use the 100-mL attenuator with the 35-mL reaction vessel.



3. Place the appropriate attenuator assembly (based on vessel size) into the cavity with the large slot positioned toward the back of the instrument and rotate clockwise until the attenuator locks into position.



100-mL Closed Vessel Reactions

Reactions can be performed under an inert atmosphere. If desired, prepare the vessel in a glovebox or pierce the cap and purge the vessel prior to performing a reaction.

- 1. Place the 100-mL vessel into the vessel stand.
- **2.** Place the attenuator on top of the vessel and stand so the top of the vessel comes through the bottom of the attenuator.
- **3.** Place the collar around the reaction vessel with the angled edge pointing downward.



- **4.** Place the cap on the vessel.
- **5.** Place the attenuator assembly into the cavity with the large slot positioned toward the back of the instrument and rotate clockwise until the attenuator locks into position.



Open Vessel Reactions

1. Place the Teflon disk flat in the cavity. Ensure the cutout in the disk aligns with the hole in the spill cup.



- 2. Place the round bottom flask with the reaction mixture and a stir bar into the cavity.
- **3.** Place the attenuator assembly into the cavity with the large slot positioned toward the back of the instrument and rotate clockwise until the attenuator locks into position. If applicable, attach extension glassware and/or condenser.



Load and Perform a Method

1. Select/Load the appropriate method. See "Methods" section or contact CEM Corporation if assistance is needed creating a new method.

NOTE

Pressure venting parameters require the Activent to relieve pressure; therefore, only 10-mL, 35-mL and 100-mL reaction vessels will implement the pressure venting parameters. Pressure venting will be ignored for all other reaction vessels.

- 2. Press the Play icon to view the run screen.
- **3.** Select the Play button to begin the method. For closed vessel reactions, the Activent will close and lock onto the vessel. The sample will heat depending on the method that was selected.
 - **3.1.** After selecting the play button select the Floating Camera button and verify that the sample is stirring.
 - **3.2.** The hotkey buttons can be utilized to edit the method parameters temperature, pressure, power, and stirring.
 - **3.3.** Images or video can be taken by selecting the camera icon.

A PINCH POINT

Never place hands into the attenuator area once play is pressed.

OCAUTION

Observe the entire synthesis process when establishing a procedure for the first time to ensure proper system functionality.

Vessel Removal and Cleanup

🛆 WARNING

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

10-mL and 35-mL Vessel

1. Once the vessel has properly cooled, the Activent will open (closed vessel reactions only) and "Idle" will appear on the display.

NOTE

If the Activent 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 Activent.

- **2.** Remove the vessel from the attenuator.
- **3.** Remove the cap from the vessel by pressing up on one side of the cap.

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

4. Proceed with work-up.

100-mL Reaction Vessel

1. Once the vessel has properly cooled, the Activent will open (closed vessel reactions only) and "Idle" will appear on the display.

NOTE

If the Activent 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 Activent.

- 2. Rotate the attenuator assembly counterclockwise and lift it from the instrument.
- 3. Place the attenuator and vessel in the vessel stand.
- **4.** Remove the cap and the collar. The collar can be removed by using any ferromagnetic (iron, nickel, steel, etc.) implement.



- 5. Lift the attenuator and slowly allow the reaction vessel to slowly slide through the bottom of the attenuator.
- 6. Proceed with work-up.

Open Vessel Reaction

- **1.** If applicable, remove the extension glassware and/or condenser.
- 2. Remove the open vessel attenuator.
- 3. Carefully remove the vessel from the cavity.
- 4. Remove the Teflon disk from the bottom of the cavity.
- 5. Proceed with work-up.

Data Management

The Discover 2.0 has a 256 GB hard drive and can store a large number of results that can be recalled by the user. In order to find specific data more easily, the users has the ability to sort and filter results based on the name, notes, result status or the time and date of analysis.

- **1.** From the Home Screen, select Results. A list of every sample that has been run will appear.
- 2. Results can be searched by entering a method name in the search field, sorting or selecting the filter icon.
- 3. Select the desired run to view further details.
- 4. If desired, a report can be generated and saved and/or results can be exported.

The Methods screen allows all users to create, edit, and delete method(s).

Creating and editing methods are independent of the currently installed vessel attenuator (vessel size and open/ sealed vessel) but dependent on the vessel type (Pyrex or Pyrex with Liner).

Method Control Types

The Discover 2.0 has five (5) control types for programming a method – Standard, Dynamic, Ramp To Temperature, Power Cycling, and Fixed Power. These control types permit the user to control how the system applies microwave energy to the reaction. In all control types, the user inputs control parameters to create the method. Only the Dynamic and Ramp To Temperature control types allow the user to program pressure venting setpoints during the reaction. The Standard control type is recommended for routine operation.



Standard

The Standard control type achieves temperature as quickly as possible and holds for desired time. All other method parameters are controlled by the instrument defaults. The power automatically adjusts based on sensor feedback to ensure the control point is reached rapidly, but with limited temperature or pressure overshoot. The user programs:

- Temperature temperature control point
- · Time the time the system maintains the control parameters

Dynamic

The Dynamic control type provides more flexibility than a standard method by allowing the user to program all method parameters. The power automatically adjusts based on sensor feedback to ensure the control point is reached rapidly, but with limited temperature or pressure overshoot. The user programs:

Stages - multiple stages can be programmed to maintain critical control points.

- Temperature temperature control point
- · Time amount of time the system maintains temperature or pressure setpoint

- Pressure maximum pressure control point
- · Power maximum amount of microwave power applied. Power will adjust to maintain control setpoint.
- Stirring stirring speed control
- Pressure venting (must be turned on in settings): Permits the user to program parameters that allows the pressure device to release unwanted gaseous byproducts from the reaction to prevent over-pressurization and vessel failure from gaseous buildup. Multiple pressure venting stages can be programmed. Pressure venting for can be turned off by deleting the Pressure Venting stage.
 - Delta Pressure the minimum assigned amount of pressure to drop from the pressure set point. A delta of 50 psi (3.5 bar) is recommended.
 - Pressure Setpoint the pressure set point at which the venting will begin.
 - If the gaseous byproduct that is generated is the only pressure in the reaction vessel (the reaction temperature does NOT exceed the boiling point of the solvent), the Activent pressure release can be set below the control point.
 - If a gas is generated, but some of the pressure is due to vapor pressure, then the release point for the Activent must be set at or above the pressure control point.
 - Times at Setpoint the maximum number of times a reaction will vent. Five (5) vents are recommended.

NOTE

Pressure venting stages operate independently of microwave heating stages.

Pressure venting parameters require the Activent to relieve pressure; therefore, only 10-mL, 35-mL and 100-mL reaction vessels will implement the pressure venting parameters. Pressure venting will be ignored for all other reaction vessels.

CAUTION

If the reaction temperature exceeds the solvent boiling point and too many venting actions are performed, the vessel could become dry and superheat.

Ramp To Temperature

The Ramp To Temperature control type allows the user to obtain temperature over a specified period of time and then hold at the temperature. The users programs:

Stages - multiple stages can be programmed to maintain critical control points.

- Temperature temperature control point
- $\cdot\,$ Ramp Time the time the reaction will take to reach the control setpoint
- $\cdot\,$ Hold time the time the system maintains the control setpoint
- Pressure maximum pressure control point
- · Power maximum amount of microwave power applied. Power will adjust to maintain control setpoint
- Stirring stirring speed control
- Pressure venting (must be turned on in settings): Permits the user to program parameters that allows the
 pressure device to release unwanted gaseous byproducts from the reaction to prevent over-pressurization and
 vessel failure from gaseous buildup. Multiple pressure venting stages can be programmed. Pressure venting for
 can be turned off by deleting the Pressure Venting stage.
 - Delta Pressure the minimum assigned amount of pressure to drop from the pressure set point. A delta of 50 psi (3.5 bar) is recommended.

- Pressure Setpoint the pressure set point at which the venting will begin.
 - If the gaseous byproduct that is generated is the only pressure in the reaction vessel (the reaction temperature does NOT exceed the boiling point of the solvent), the Activent pressure release can be set below the control point.
 - If a gas is generated, but some of the pressure is due to vapor pressure, then the release point for the Activent must be set at or above the pressure control point.
- Times at Setpoint the maximum number of times a reaction will vent. Five (5) vents are recommended.

NOTE

Pressure venting stages operate independently of microwave heating stages.

Pressure venting parameters require the Activent to relieve pressure; therefore, only 10-mL, 35-mL and 100-mL reaction vessels will implement the pressure venting parameters. Pressure venting will be ignored for all other reaction vessels.

CAUTION

If the reaction temperature exceeds the solvent boiling point and too many venting actions are performed, the vessel could become dry and superheat.

Power Cycling

The Power Cycling control type allows the user to apply the desired power to the reaction until a power interval time or temperature maximum is reached. Irradiation then ceases and the reaction cools until the cooling interval time or minimum temperature is reached. This cycle is repeated for a user-defined number of times. The user programs:

- · Power maximum amount of microwave power applied
- · Power Interval the maximum amount of time microwave power is applied
- · Cooling Interval the maximum amount of time microwave power is turned off
- · Maximum Temperature maximum temperature (power interval)
- · Minimum Temperature minimum temperature (cooling interval)
- · Number of Cycles the number of power/cooling cycle repetitions that will be completed

Fixed Power

The Fixed Power control type allows the user to apply the desired power from the beginning of the reaction until the temperature is reached. It provides the user the most direct method to energize reaction systems. The user programs:

- Power amount of constant microwave power applied
- Time maximum run time (the total time the system applies microwave energy)
- Temperature maximum temperature (a temperature above which the system will not apply microwave energy)
- Temperature type:
 - Control Fixed applies programmed power until the temperature setpoint is reached, then adjusts power to
 maintain the temperature setpoint
 - Safe applies programmed power until the end of the programmed reaction time or until the temperature setpoint is reached
- Stirring stirring speed control

Create Method

- **1.** Select "Methods" from the Home Screen.
- **2.** Select "New Method."

New me	thod	25°C	STAC TIN POWE PRESSUR	3E Ke ER Re	
Info Method				Can b	2
Basic information about the method					
NAME	New method				
TAGS	(P)				
CREATED BY	CEM Administrators				
LAST USED	Never				
LAST MODIFIED	Never				
NOTES					

- **3.** Enter a unique method name.
- **4.** If desired, add a tag and/or notes.
- 5. Select the "Method" tab.

New meth	nod			25°C	STA TI POW PRESSU	ige Me Jre Jre		- ×
Info Method						Can t	2	Ŵ
VESSEL TYPE	Pyrex	•						
CONTROL TYPE	Standard	•						
TEMPERATURE		100	°C					
TIME		00:05:00						

- 6. Select and choose the "Vessel Type."
- 7. Select and choose the "Control Type." Method parameters will vary depending on the "Control Type" selected.
- **8.** Enter the remaining parameters.
- **9.** If applicable, select the "Stages" tab and enter the desired parameters. "Stages" are only available for the Ramp to Temperature or Dynamic control types.
- **10.** If applicable, select the "Pressure Venting" tab and enter the desired parameters. "Pressure Venting" is only available for Dynamic and Ramp To Temperature control types.
- **11.** Select the "Save" icon in the top left corner of the screen.

CAUTION

Observe the entire synthesis process when establishing a procedure for the first time to ensure proper system functionality.

Edit Method

- **1.** Select "Methods" from the Home Screen.
- **2.** Select the method to be edited.
- **3.** Select the Edit icon on the right side of the screen.
- 4. Select desired tab to edit (Method, Stages, or Pressure Venting) and edit the parameters.
- 5. Select the Save icon located in the top left corner of the screen.

Delete Method

- 1. Select "Methods" from the Home Screen.
- 2. Select the method to be deleted.
- 3. Select the Delete icon on the right side of the screen.
- 4. Select "Remove" to delete the method or back to return to the previous screen.

Export Method

- 1. Select "Methods" from the Home Screen.
- 2. Select "Import/Export" located in the lower right corner of the screen.
- 3. Select "Export Methods."
- **4.** Select the method(s) to export.
- **5.** Select "Export ____ Method(s)" to export the desired methods or "Cancel" to return to the previous screen.
- 6. If "Export" is selected, select the location and save.

Import Method

- **1.** Select "Methods" from the Home Screen.
- 2. Select "Import/Export" located in the lower right corner of the screen.
- 3. Select "Import Methods."
- 4. Locate the method(s) to import and select "select."

Users

The Users menu allows control of Discover 2.0 instrument access based on locally created accounts or a network domain. Enabling a local user type prompts creation of an initial administrator account who can add additional users to one of three default user groups: Administrators, Users, or Guests.

DISCOVER	- *	DISCOVER	- ×
✓ Users		• Users	
Users On/Off > Select user type		Users On/Off > Select user type	
Data Integrity Adjust data integrity and traceability settings		Edit Users Create and edit system user > accounts	
		Edit User Groups Create and edit system user > groups	
		Login/Password Adjust login and password requirements	
		Data Integrity Adjust data integrity and traceability settings	

Edit Users

The Edit Users menu allows management of Discover 2.0 users by adding, editing, deactivating, or hiding user accounts.

Edit User Groups

The User Groups menu defines the classification of user accounts based on permissions and priority for system access. This menu allows creation of new groups, removal of existing groups, and editing of group permissions and priorities.

Login/Password

The Login/Password menu increases or decreases security requirements for the title categories.

Data Integrity

The Data Integrity menu implements additional approval requirements to provide improved audit trail traceability.

Software Update

The current version of Discover 2.0 software can be found on the CEM website. Please register the Discover 2.0 and create an account to access software updates.

How to Update Software

- **1.** Download the Discover 2.0 software and copy onto USB stick on a directory that can be accessed on the Discover 2.0
- 2. If applicable, insert the USB Stick containing the software update bundle into the Discover 2.0 USB port.
- 3. Select the System Menu icon.
- **4.** Select Settings -> Configuration -> Update System-> Update Software.



- 5. Select the appropriate file path to access the new software followed by "Select."
- 6. The software will automatically install.

System Repacking

Follow procedures below to prepare the Discover 2.0 for shipment.

Prepare to Ship

- **1.** Ensure the system is idle.
- **2.** Prepare the shipping device.
 - **2.1.** Remove the thumb screen from the shipping device.
 - **2.2.** Ensure the shipping device has a cap and place the shipping device into the 35-mL attenuator.
 - **2.3.** Use the thumb screw to secure the shipping device in the attenuator.
 - **2.4.** Place the 35-mL attenuator with shipping device into the microwave cavity and turn clockwise to lock in place.
- **3.** Close the Activent on the shipping device by selecting the System Menu -> Tools -> Diagnostics -> Diagnostics -> Activent Close and then select the play icon.
- **4.** After the Activent has sealed onto the shipping device turn the instrument off using the power switch located on the left side of the unit.

Never ship the unit without the shipping device in position and the Activent pressure device in the closed position.

- 5. Disconnect any peripherals (Autosampler, Gas Addition, Flow Cell or CoolMate).
- 6. Disconnect the power cord and place into the power cord box.
- **7.** Disconnect the following items and place in the accessory kit: Vent Line tubing, air regulator with tubing, and any remaining accessories in the accessory kit.
- 8. If accessible, wrap the plastic sheet around the Discover 2.0.
- 9. Place the foam pieces around the Discover 2.0, ensuring that the cutouts fit around the system.
- **10.** Carefully place the instrument into the shipping box.
- **11.** Place the power cord box into the cutouts in the foam.
- 12. Place the accessory kit on top of the foam. DO NOT SHIP without an accessory kit in place.
- **13.** Close the box and place tape along the top and top/side seams.

Maintenance Overview

Annually

Preventative Maintenance by CEM Certified Technician

This is performed by a CEM Certified Technician, once per year. However, should your individual requirements vary, a CEM Representative is available to assist in creating a schedule to meet your needs. Contact the CEM Contract Administrator at 800-726-3331 for assistance.

As Needed

- · Clean the interlocks
- Clean the cavity and iWave Sensor
- \cdot Clean the spill tray
- Water Verification
- iWave Temperature Calibration
- Activent Pressure Calibration

Maintenance Procedures

CEM suggests that you perform the routine maintenance procedures as outlined below. See the "Maintenance Overview" section for maintenance schedules.

A complete listing of spare and replacement parts with part numbers and images can be found on the CEM website (www.cem.com).

Interlock Cleaning

The interlocks needs to be cleaned/inspected if a vessel failure occurs.

- **1.** Ensure the system is idle.
- **2.** Turn the Discover 2.0 power switch off and disconnect the power cord.
- 3. If applicable, remove the attenuator by rotating counterclockwise. This will expose the interlocks and cavity.
- 4. Inspect the interlocks and verify that they are clean and free of debris.
- 5. If the interlock appears dirty, use a cotton swab and methanol, ethanol, or acetone to clean.



- 6. Once the solvent is dry the attenuator can be installed.
- 7. Install the power cord.
- 8. Turn the instrument on and continue routine operation.

NOTE

If attenuator becomes difficult to turn or cannot be locked into position, please contact CEM Service.

Cavity, Spill Cup and iWave Cleaning

The cavity needs to be cleaned/inspected if a vessel failure occurs or material is spilled in the cavity. If any damage is noted to components, contact the service department at CEM Corporation.

- **1.** Ensure the system is idle.
- 2. Turn the Discover 2.0 power switch off and disconnect the power cord.
- 3. If applicable, remove the attenuator by rotating counterclockwise. This will expose the interlocks and cavity.
- 4. Remove the spill cup by pulling straight up. The spill cup protects the temperature sensor from debris in case

of a vessel failure.



- **5.** Clean the spill cup by rinsing with water, ethanol, methanol, or acetone. If necessary, the cup can be wiped with a cloth. Once all debris is rinsed from the cup, check for cracks and/or damage. Discard and replace the spill cup if damage is noted.
- 6. Wipe the cavity liner with an alcohol wipe or equivalent.
- **7.** Inspect the cavity liner for foreign debris or charring. If charring is seen on the cavity liner, discontinue use and contact CEM Corporation.



- **8.** Inspect the iWave temperature sensor and camera lens for foreign debris and/or damage. If necessary, use a swab and alcohol to carefully clean the lens, ensuring not to scratch it. If the lens appears to be damaged (ie. scratched), discontinue use and contact CEM Corporation.
- 9. If the spill cup is not damaged, replace the spill cup once it has dried.
- 10. Install the power cord.
- **11.** Turn the instrument on and continue routine operation.

Spill Tray Cleaning

The spill tray needs to be cleaned/inspected if a vessel failure occurs or material is spilled in the cavity.

- **1.** Ensure the system is idle.
- **2.** Turn the Discover 2.0 power switch off and disconnect the power cord.

- **3.** Turn the instrument so that the back of the system is visible and accessible.
- 4. Remove the screws securing the spill tray to the back of the instrument cover.



5. Carefully slide the spill tray out of the instrument.



- 6. Properly dispose of the contents of the tray.
- **7.** If necessary, rinse the tray with an appropriate solvent and allow to dry. The filter can be removed, cleaned and positioned in the spill tray once dried.



- 8. Install the spill tray and secure it to the instrument with the thumb screws.
- 9. Install the power cord.
- **10.** Position the instrument so it is facing forward and the display and power switch are easily accessible.
- **11.** Turn the instrument on and continue routine operation.

Water Verification

The "WATER VERIFICATION TEST" method can be used as a check to ensure temperature and pressure measurement are operating correctly, but is not necessarily an indication as to which is erroneous.

- 1. Place 5 mL of tap water in 10-mL vessel or 20 mL of water into a 35-mL vessel.
- 2. Add an appropriately sized stir bar into the vessel and place the cap on the vessel.
- 3. Place the vessel into the microwave cavity.
- 4. Load the "Water Verification Test" method. If the "WATER VERIFICATION TEST" is not available, then program a method using the following parameters: Control Type = dynamic; Temperature = 200 °C; Time = 2:00; Pressure = 250 psi; Power = 300, and Stirring= high.
- **5.** Select the Play icon to begin the run.
- **6.** The sample should heat to 200 °C and 200 psi (± 10%). If the sample is not within 10% contact CEM Corporation.

iWave Temperature Calibration

- 1. Select the System Menu icon.
- 2. Select Tools -> Calibrations -> Calibration -> Temperature -> iWave.



- **3.** Select the appropriate "Vessel Type" followed by the Play icon.
- 4. Follow the instructions on the screen to complete the iWave temperature calibration.

Activent Pressure Calibration

- **1.** Select the System Menu icon.
- 2. Select Tools -> Calibrations -> Calibration -> Pressure > Activent Pressure.



- 3. Select the appropriate "Vessel Type" followed by the Play icon.
- 4. Follow the instructions on the screen to complete the Activent pressure calibration.

Problems and Possible Solutions

Problems	Possible Cause	Possible Solutions
Reaction yield/result changes despite identical reaction conditions	iWave requires calibration	Perform the "water verification" test to verify that reported pressure is within \pm 10% of 200 psi (13.8 bar). If pressure is outside of the acceptable range, perform the "iWave calibration" followed by the "Activent calibration" procedure.
	Stirring is not adequate	Use a large, rare earth stir bar and verify stirring with the camera
Target method temperature cannot be reached	Power setting is too low	Increase method power setting in 50 or 100 W increments until maximum power is reached
	Stirring is not adequate	Use a large, rare earth stir bar and verify stirring with the camera
	Sample does not absorb microwave energy	Perform reaction at a higher concentration, switch to a better microwave absorbing solvent, or use a silicon carbide vessel
	PowerMAX is enabled	Turn off PowerMAX in the problematic heating stage
Erratic pressure readings or over-pressurization during run	Reusing vessel caps leading to inaccurate pressure measurement	Ensure new caps are used for every reaction
	Stirring is not adequate	Use a large, rare earth stir bar and verify stirring with the camera
	Heating reaction beyond instrument pressure limits	Reduce reaction temperature by 25 °C and retry synthesis
	Reaction evolves a gaseous by- product	Use Activent to relieve gas pressure
Pressure device will not release reaction upon completion	Release limits are not met	Verify that cooling air valves (source and regulator) are open and air flow can be heard
	Reusing vessel caps leading to inaccurate pressure measurement	Ensure new caps are used for every reaction
	Residual pressure from gaseous by-products	If the reaction temperature is below the reflux temperature of the solution, manually release the vessel
Vessel Failure	Contact CEM Corporation	Perform all procedures in the "Maintenance Procedures" section

Create a Troubleshooting bundle

A troubleshooting bundle can be submitted to CEM Corporation for system analysis and troubleshooting. To create a troubleshooting bundle:

- **1.** Select the System Menu icon.
- 2. Select Help -> Create Troubleshooting Bundle -> "Create Troubleshooting Bundle" button.
- **3.** Selection the file location and the Select button.
- 4. The .zip folder can be submitted to CEM Corporation for analysis.

Location Requirements

- Designated area with a sturdy workbench capable of supporting system weight and optional accessories
 The system weighs 38 lbs (17.3 kg)
- Provides adequate space for sample handling
- $\boldsymbol{\cdot}$ Is free from vibration of large equipment
- · Provides clear visual access to the display of the system
- · Compressed Gas Source Any compressed air, nitrogen, or argon source with the following specifications
 - Within eight feet (8') of the reactor
 - · Capable of supplying a minimum of 25 psi and a maximum of 60 psi

Instrument Dimensions

- Discover 2.0 Instrument Dimensions
 - Width: 14" (35.6 cm)
 - · Depth: 18.5" (47 cm)
 - Height: 11" (28 cm)
- Recommended Counter Space:
 - Width: 30" (76.2 cm) includes required 8" (20 cm) of open space on both sides of the system
 - Depth: 24.5" (62.3 cm) includes required 6" (16 cm) of open space behind the instrument for ventilation
 Height: 11" (28 cm)
- Do not position the Discover 2.0 so it is difficult to access the power switch
- Optional Accessories: An additional eight (8) inches (20 cm) of open space required on both sides of the system and six (6) inches (16 cm) of open space required behind the instrument for ventilation.
 - Autosampler with Discover 2.0 Dimensions
 - 16" (40.7 cm) W x 18.5" (47 cm) D x 32.5" (82.6 cm) H
 - Gas addition with Discover 2.0 Dimensions
 - 20" (50.8 cm) W x 18.5" (47 cm) D x 11" (27.9 cm) H
 - Must be directly beside Discover 2.0
 - CoolMate with Discover 2.0 Dimensions
 - + 31" (78.7 cm) W x 18.5" (47 cm) D x 13.1" (33.3 cm) H
 - · Avoid shelves or cabinetry above unit so cooling fluid and dry ice or liquid nitrogen can be added.
 - Must be directly beside Discover 2.0

Environmental Requirements

- Indoor use only
- Altitude up to 2000 m
- Operating temperature range of 50 °F (10 °C) to 85 °F (29.4 °C)
- Relative humidity range of 10 % to 85 %, non-condensing
- Overvoltage Category II
- Pollution degree 2

Electrical Requirements

- Discover 2.0 is Region Specific
 - 100 120 VAC, 50/60 Hz, 10 A or
 - · 220 240 VAC, 50/60 Hz, 5 A
 - Fuse Replacements:
 - 100 200 V, 50/60 Hz: 250 V, 10 A 6.3 mm x 32 mm, fast acting
 - 220 240 V, 50/60 Hz: 250 V, 5 A 6.3 mm x 32 mm, fast acting
- Autosampler 12 & 48
 - 100 240 VAC, 50/60 Hz: 250 V, 2 A
 - Fuse Replacements:
 - 100 240 V, 50/60 Hz: 250 V, 2 A 5 mm x 20 mm, fast acting
- \cdot Line voltage must not vary more than ±10% of its specified level
- Dedicated, grounded outlet
- · Instrument is supplied with a power cord or an adequately rated power cord may be used

Limited Warranty Information

What Is Covered:

CEM Corporation warrants that the instrument will be free of any defect in parts or workmanship and will, at its option, replace or repair any defective part (excluding consumables) or instrument.

For How Long:

This warranty remains in effect for 365 days from date of delivery to the original purchaser.

What Is Not Covered:

This warranty does not cover parts or workmanship damaged due to:

- Neglect, abuse or misuse,
- Damage caused by or to test samples,
- Damage incurred during instrument relocation,
- Damage caused by or to any attached equipment,
- Use of incorrect line voltages or fuses,
- Fire, flood, "acts of God" or other contingencies beyond the control of CEM Corporation,
- · Improper or unauthorized repair, or
- Any other damage caused by purchaser or its agents.

Responsibilities of Purchaser:

To ensure warranty coverage, the purchaser must:

- · Use the instrument according to directions,
- · Connect the instrument properly to a power supply of proper voltage,
- · Replace blown fuses,
- · Replace consumables and
- · Clean the instrument as required.

How to Get Service:

Purchaser should contact the Service Department of CEM Corporation or the nearest CEM subsidiary or distributor for return authorization and for proper crating and shipping instructions to return instrument, freight prepaid, for service. On-site repairs by an authorized service technician are available through the CEM Service Department. Travel costs will be charged to the purchaser for on-site repairs.

Within the U.S.	Outside the U.S.
CEM Corporation	CEM Corporation
3100 Smith Farm Rd.	3100 Smith Farm Rd.
Matthews, NC 28105-5044	Matthews, NC 28105-5044
(800) 726-5551	(704) 821-7015
Fax: (704) 821-4368	Fax: (704) 821-4368

Warranty Disclaimer:

CEM Corporation hereby excludes and disclaims any warranty of merchantability or fitness for any particular purpose. No warranty, express or implied, extends beyond the face hereof. CEM Corporation shall not be liable for loss of use of instrument or other incidental or consequential costs, expenses or damages incurred by the purchaser or any other user. This warranty is not transferable.

Purchaser's Rights under State Law:

This warranty gives the purchaser specific legal rights, and the purchaser may also have other rights that vary from state to state.

Applications Support

For the latest Discover 2.0 applications information, go to http://cem.com/Discover-2/. The Discover 2.0 pages contain downloadable applications notes, a listing of recent posters, method notes and more. CEM is proud to provide applications support for any synthesis related questions from a team of trained chemists with a complete synthesis lab. For applications support, call (800) 726-3331 (inside the US) or (704) 821-7015 and ask for "Synthesis Support", or email Discover 2.0 applications support at synthesis.support@cem.com.

Technical Support

CEM is proud to provide technical support for the Discover 2.0 from a team of specially trained Service Technicians. For technical support in the US and Canada, call (800) 726-5551 or (704) 821-7015 and ask for "Discover 2.0 Service" or email service@cem.com. For technical support outside the US and Canada, contact your local CEM Subsidiary or Distributor.

Requested Information

When contacting CEM for support, please provide the following information about the instrument:

- Discover 2.0 Serial Number
- Discover 2.0 Software Version
- Troubleshooting bundle

The serial number, software version and troubleshooting bundle can be accessed from the Help menu.

Service can only be performed by an authorized CEM service technician.

CEM Corporation Headquarters

Toll-Free Phone (US/Canada): (800) 726-3331 Phone: (704) 821-7015 Service Hotline: (800) 726-5551 Fax: (704) 821-7894 Mailing Address: PO Box 200 Matthews, NC 28106-0200 Physical Address: 3100 Smith Farm Rd Matthews, NC 28104 Email (Applications Support): synthesis.support@cem.com Email (Technical Support): service@cem.com

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Phone: 390-35-896224 Fax: 390-35-891661 Address: Via Dell Artigianato, 6/8 24055 Cologno Al Serio (BG) Italy Web Address: http://www.cemmicroonde.it Email: info.srl@cem.com

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CEM Distributors

For a complete list of distributors of CEM products, including contact information, go to the CEM website (http:// www.cem.com), select Contact, and then select your region to see a list of distributors by country.

CEM

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