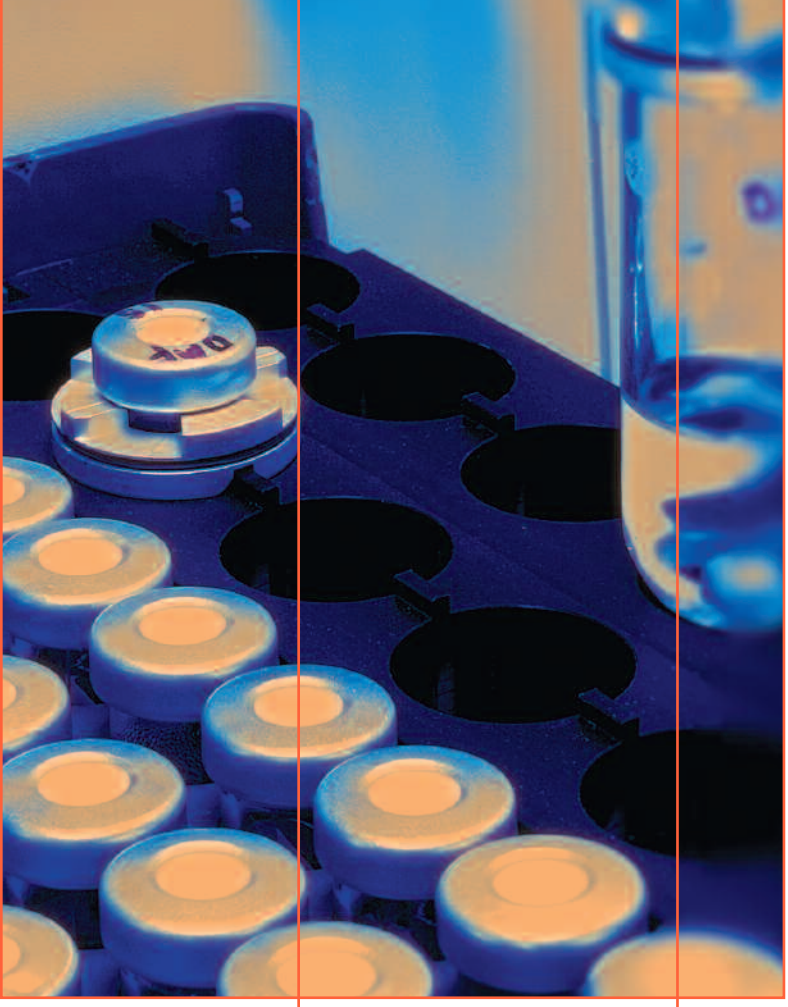


Initiator™

GETTING STARTED GUIDE

www.biotage.com

Biotage

Table of contents

Quick start

Preparing your experiment

1. Dispense the chemicals into an unused microwave vial and insert a magnetic stirring bar. Do not fill the microwave vial above or under the stated volume range, and make sure that solid material is not present on the walls.

2. Cap the microwave vial properly. The cap should clasp the vial brim or the vial adapter and not be possible to turn by hand. The top of the cap should be flat.

Note: If you are using 0.2-0.5 mL or 10-20 mL microwave vials, use vial adapters. You need to position the vial adapter before capping the 0.2-0.5 mL vial. (These vials can only be used with EXP systems.)

The vial to the right is capped too tightly.

Warning: Observe general as well as specific safety regulations for the use of the equipment and its accessories and consumables at all times, in order to reduce the risk of personal injury, fire and explosion, see the *Warning summary* in the *Installation & Safety* document.

Set up user accounts

A user can have system owner and/or chemist privilege:

- The chemist privilege gives the user access to the chemistry mode, i.e. the user can set up and run experiments, and view experiment results.

- The system owner privilege gives the user access to the system mode, i.e. the user can change system settings, manage users and calibrate the touch screen. It is possible to password protect a user account with system owner privilege.

Note: User accounts can only be set up when the instrument is not processing and by a user with system owner privilege. If your company does not have a user with system owner privilege, use the account that was created at installation (to find out the password, see *Installation* in the *Installation & Safety* document).

To add a new user:
Note: This can be done only by users with system owner privilege.

1. If you are not in the main menu, press **Main Menu** in the right-hand panel.

2. Press **System**. The **Select User** dialog appears.

3. Select user and press **OK**. The **Input Password** dialog appears.

4. Enter password and press **OK**.

5. Select the **Manage Users** tab.

6. **Add** a new user account. For detailed information, press **Help**.

7. To save the new account, press **Save**.

8. Press **Log Out** to return to the main menu.
To set up and run experiments, press **Chemistry**.

Set up and run your experiment on Initiator (without Robot)

1. Select the **Editor** tab in the right-hand panel.

2. Edit the process parameters. To edit time, temperature and/or pre-stirring, press the parameter's button, enter the value on the keypad to the right and then press **Enter**. To change vial type, absorption level and/or whether fixed hold time is used or not, repeatedly press the parameter's button until the desired value is displayed.

3. Press **Run**.

4. When the **Load Experiment** dialog appears, insert the microwave vial into the microwave cavity.
Note: In EXP systems, the cavity insert must be manually removed from the microwave cavity before loading Biotage Microwave Vials 10-20 mL, and manually inserted before loading Biotage Microwave Vials 0.2-0.5 mL, 0.5-2 mL or 2-5 mL.

5. Select user and enter the experiment name in the **Load Experiment** dialog.

6. To confirm loading and start the experiment, press **Run**.

Set up and run your experiment on Initiator (with Robot)

1. Select the **Editor** tab in the right-hand panel.

2. Enter the number of reactions by pressing **Number of Vials**. You can also add a reaction to the experiment by selecting an empty vial position in the rack overview.

3. To edit a reaction's process parameters or several reactions simultaneously, select the vial or vials in the rack overview and press **Edit**. The **Edit** dialog appears. To edit time, temperature and/or pre-stirring, press the parameter's button, enter the value on the keypad to the right and then press **Enter**. To change vial type, absorption level and/or whether fixed hold time is used or not, repeatedly press the parameter's button until the desired value is displayed. To save the changes and return to the **Editor** tab, press **Apply**.

4. Press **Run**. If the instrument is processing, the experiment is added to the queue and the processing is paused for loading.

5. When the **Load Experiment** dialog appears, load the vial(s) into the rack according to the yellow position(s) in the rack overview.

6. Select user and enter the experiment name in the **Load Experiment** dialog.

7. To confirm loading and start/resume processing, press **Run**.

*For more information on how to set up and run experiments, see the online help, i.e. press **Help**.*

Monitor your experiment and view results

To monitor the reaction in progress:
Select the **Status** tab in the right-hand panel. With the **Show Values**/**Show Graph** button, you can toggle between viewing:

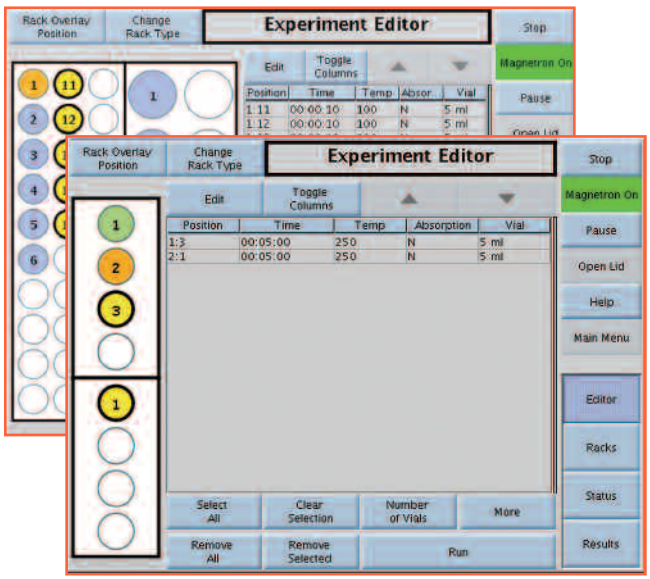
- The process graph with real time measurements of temperature, pressure and applied power.
- The target values (temperature, pressure and/or power) are shown as dashed lines.
- The current values of temperature, pressure and applied power.

To view the results of your experiment:
1. Select the **Results** tab in the right-hand panel.

2. Press **Select User**. The **Select User** dialog appears.

3. Select user and press **OK**.

4. To view the results of an experiment, select the experiment in the experiment list. To scroll the list of experiments or the report up or down, press ▲ or ▼.



*The color of the vial position indicates the processing status. Press **Help** for color legend.*

Instrument overview

A = touch screen, B = main switch, C = cavity lid, D = cavity insert located in the insert holder, E = robot, F = sampler arm, G = gripper and H = rack.

The Initiator system can process reaction volumes between 0.5 and 5 mL. The instrument is equipped with a touch screen used for experimental planning, instrument control and reaction monitoring. The instrument status is displayed on the touch screen's right-hand panel:

- Idle or Paused = The instrument is not processing.
- Processing = The instrument is processing, but the magnetron is switched off.
- Magnetron On = The instrument is processing and the magnetron, which generates microwaves, is switched on.

Heating, stirring and cooling

When the microwave vial has been inserted into the microwave cavity and the cavity lid has been closed, high-frequency microwaves (2.45 GHz), generated by the magnetron, heat the reaction mixture.

During the heating process, the reaction mixture is continuously stirred by means of magnetic stirring. If stirring is unwanted the magnetic stirring bar is simply omitted. It is also possible to stir the reaction mixture before the heating process is started to swirl up the content to improve the microwave absorption optimization and avoid large aggregates of solids that might otherwise cause vial breakage.

After processing, the reaction mixture is immediately cooled with pressurized air. When the temperature of the reaction mixture has dropped to 40 °C or 50 °C (according to your system setting), the cavity lid is opened and the microwave vial can be removed.

Robot automation

If your system is equipped with a robot, all experiments are processed through a queue and the transportation of the vials is handled by the system. This allows new experiments to be planned and added to the queue as long as there are free positions in the racks. Depending on the robot size, the queue can contain a maximum of 8 or 60 microwave vials.

There are two robot sizes available, Eight and Sixty. Robot Eight can handle vial racks with 2 or 4 positions, Robot Sixty can handle vial racks with 12 or 30 positions. The 2 position accommodates vial sizes 10-20 mL (with EXP function). The 4 position accommodates vial sizes 0.5-2 and 2-5 mL. The 12 position accommodates vial sizes 10-20 mL (with EXP function) and the 30 position accommodates vial sizes 0.5-2 and 2-5 mL.

Expanded volume range (EXP)

With EXP systems, the working volume range is expanded from 0.5-5 mL to 0.2-20 mL. The use of a cavity insert allows you to freely choose between four different vial sizes; 0.2-0.5 mL, 0.5-2 mL, 2-5 mL and 10-20 mL.

The cavity insert must be manually removed from the microwave cavity before loading 10-20 mL vials and manually inserted before loading 0.2-0.5 mL, 0.5-2 mL or 2-5 mL vials. If the system is equipped with a robot, the cavity insert is automatically inserted and removed by the gripper and sampler arm.

Waste tray

If, for some reason, a microwave vial breaks or leaks in the microwave cavity, the glass and the reaction mixture are collected in the waste tray underneath the microwave cavity. The waste tray can be removed and cleaned as described in the maintenance section beginning on page 13.

Software modes

- **Chemistry**: Set up and run experiments, and view experiment results.
- **System**: Change system settings, manage users and calibrate the touch screen. Only users with system owner privilege can perform these tasks.
- **Service**: Service can only be performed by an authorized Biotage service technician.
- **Shut Down**: Shut down the system.

Set up and run experiments

Depending on whether the system is equipped with a robot or not, the chemistry mode consists of 3 or 4 tabs: Editor (1), Racks (2), Status (3) and Results (4).

1. Editor

Here you can set up and run experiments. The reactions can be performed within a temperature range of 60 to 250 °C at pressures of up to 20 bar.

If your system is equipped with a robot, enter the number of reactions by pressing **Number of Vials**. You can also add a reaction to your experiment by selecting an empty vial position in the rack overview. The reactions can be edited one by one or simultaneously; select the vial or vials that you want to edit in the rack overview and press **Edit**.

To edit time, temperature and/or pre-stirring, press the parameter's button, enter the value on the keypad to the right and then press **Enter**. To change vial type, absorption level and/or whether fixed hold time is used or not, repeatedly press the parameter's button until the desired value is displayed.

To perform the experiment, press **Run**. When the **Load Experiment** dialog appears, select user, enter experiment name and load your experiment into the instrument. Confirm loading by pressing **Run**. The heating process in progress can be monitored at the **Status** tab.

Three different absorption levels are available: **Normal**, **High** and **Very High**. The high or the very high level should be used when heating reaction mixtures that include very polar solvents or have high ionic content, e.g. containing inorganic acids or ionic liquids. In these cases, the energy is applied at a lower rate to the reaction mixture in order to achieve a well-controlled rate of temperature increase.

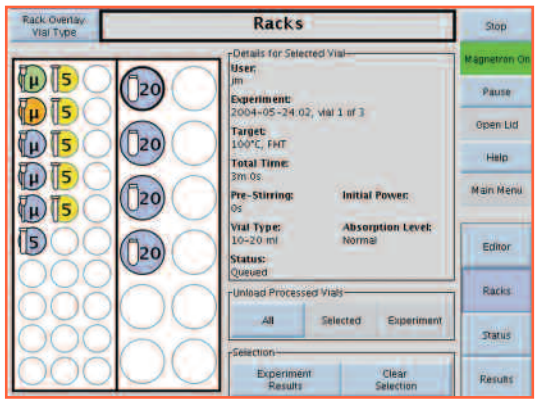
If **Fixed Hold Time** is **On**, the time countdown starts when the target temperature or target pressure is reached, i.e. the initial time taken to reach the set temperature or pressure is not included in the heating time. If **Fixed Hold Time** is **Off**, the time countdown starts when the heating starts.

If you wish to use advanced process parameters for a reaction (i.e. set up a reaction including several heating or cooling steps, control by means of temperature, pressure and/or power, set initial power and/or cool while heating), press **Advanced Edit**.

2. Racks

At the **Racks** tab you can view the contents of the rack(s) loaded onto the instrument and unload processed vials.

Note: This tab is only available for systems equipped with a robot.



When using Initiator Sixty EXP, you can use vial racks with 12 or 30 positions.

Software overview

If you select a vial in the rack overview, you will find out the user, experiment name, process parameters, status and, if the reaction failed (red), the reason for failure. The color of the vial position indicates the processing status; see color legend in the online help.

For example, to unload an experiment and clear the vial positions in the software, select a processed vial included in the experiment and press **Experiment**. If the instrument is processing, the processing will be paused for unloading.

3. Status
At the **Status** tab you can monitor and edit the reaction in progress, delete queued experiments and change the processing order for queued experiments.*

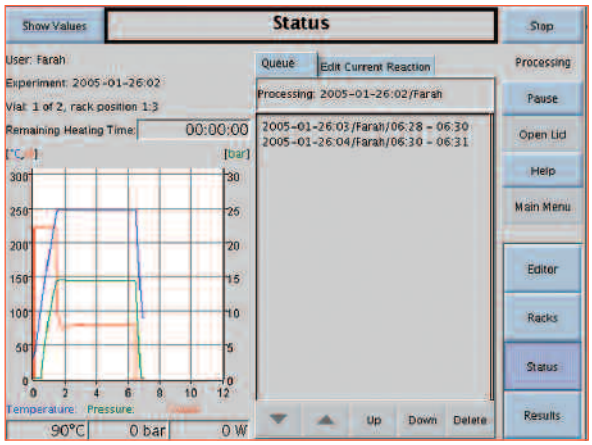
To edit the reaction in progress, select the reaction or, if the reaction consists of several steps, the step that you wish to edit. Edit a process parameter by pressing the corresponding text box. To apply your changes, press **Apply**. The changes will not be saved as set values. The resulting changes will, however, show in the results graph.

All experiments to be performed are listed in processing order in the queue list. It is possible to change the processing order and to delete a queued experiment.*

** The queue is only available for systems equipped with a robot.*

4. Results
At the **Results** tab you can view and delete the results of your experiments. **Tip!** You can view and print the results from your office through a web browser, see next section.

Online help
For more information, see the online help.



*At the **Status** tab you can monitor and edit the reaction in progress.*

View instrument status and results from your office

With the instrument connected to your network, several simultaneous users can access the Initiator Remote Viewer through a standard web browser.

The Initiator Remote Viewer quickly tells you:

- Is this instrument occupied and if so, when is it available?
- What is the status and results of my experiment(s)?
- Is the instrument still processing or is there a problem that caused the processing to stop, requiring manual attention?

To access the Initiator Remote Viewer:
1. Enter the URL *http://MACHINENAME* in a web browser (where MACHINENAME is the network identity of the instrument, or an IP-address).

2. Press **Enter** and the instrument status web page is loaded. The page is automatically updated every five seconds.

Note: If you need help accessing the Remote Viewer, contact your IT administrator.

www.biotagepathfinder.com

Biotage PathFinder is a web-based service featuring a unique microwave synthesis database including more than 3600 carefully selected microwave reactions. PathFinder gives you direct access to years of experience in microwave synthesis delivered in a detailed, easy to use and easily accessible format.

Most content has been conducted on Biotage microwave systems making the methods highly reproducible. PathFinder also includes other valuable tools, such as the "Ask-a-Chemist" feature, which provides a resource for chemists to have a dialog about microwave synthesis methods directly with a Biotage Chemist experienced in microwave synthesis. Additional features include a gas-pressure calculator and the Biotage PathFinder Cookbook.

Search the reaction database

When you log on to www.biotagepathfinder.com, the search page appears. You can search the reaction database by substructure or by reaction keyword. (Figure 1.)

The hits will be listed in an overview including reaction scheme, temperature, time, and yield or purity. (Figure 2.)

For each reaction you can view a report including all information needed to repeat the reaction. It also includes literature references, analysis results and comments about the reaction and the work-up procedure. (Figure 3.)

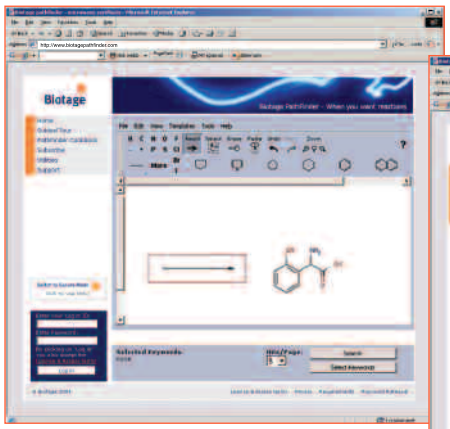


Figure 1. *Enter a simple substructure search*

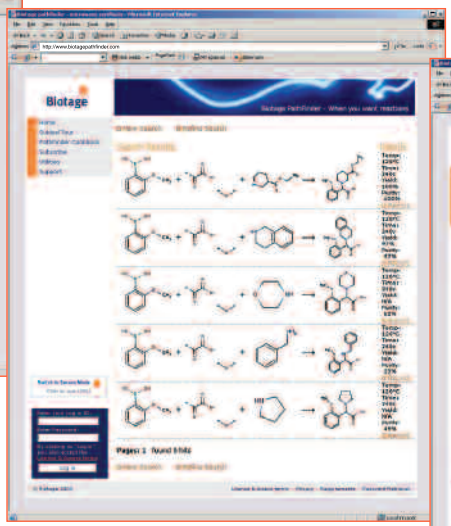


Figure 2. *Search results are displayed in an easy to browse format*

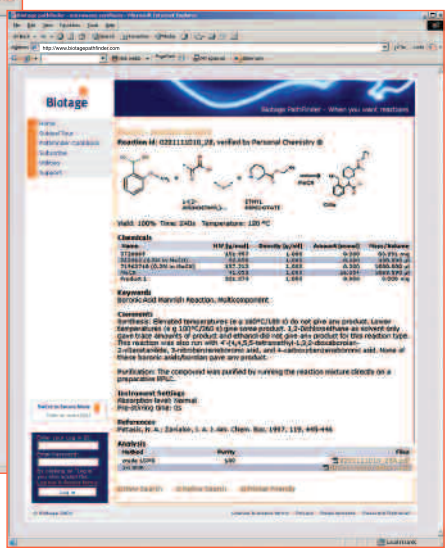


Figure 3. *Reaction details needed to perform repeated reactions are listed in an easy-to-read format*

Getting started with microwave synthesis

Microwave synthesis is normally conducted under conditions that vary considerably from what is conventionally used in today's chemistry laboratories. Although microwave synthesis often renders results that are unique the outcome is largely governed by a few, well-known phenomena. With knowledge about these phenomena, your benefits of using microwave synthesis will be greatly enhanced.

What conditions are appropriate when performing microwave synthesis?

Biotage microwave systems support a wide variety of different reaction conditions, accommodating different solvents, volumes, concentrations and phases and are characterized by reproducible results.

Solvent

- Different solvents interact very differently with microwaves, because of their diverse polar and ionic properties.
- Acetonitrile, DMF, and alcohols are often used for microwave-assisted organic synthesis.
- You might not need to change from the solvent that is specified for the reaction under traditional chemistry conditions. First, try using the solvent that you would normally use.
- Polar solvents (e.g. DMF, NMP, DMSO, methanol, ethanol, and acetic acid) work well with microwaves due to their polarity, you can be sure that the temperature will rise substantially with these solvents.
- Non-polar solvents (e.g. toluene, dioxane, THF) can be heated only if other components in the reaction mixture respond to microwave energy, if the reaction mixture contains either polar reactants or ions. When using less polar solvents, more concentrated reaction mixtures might be preferable. Under these circumstances, a very high temperature can be achieved.
- Ionic liquids are reported as new, environmentally friendly, recyclable alternatives to dipolar aprotic solvents for organic synthesis. The dielectric properties of ionic liquids make them highly suitable for use as solvents or additives in microwave-assisted organic synthesis. Ionic liquids consist entirely of ions and therefore absorb microwave irradiation extremely efficiently. Furthermore, they have a low vapor pressure, enhancing their suitability even further. Despite ionic liquids being salts, they dissolve in a wide range of organic solvents, and can therefore be used to increase the microwave absorption of low absorbing reaction mixtures.
- Solvents can behave differently at elevated temperatures and most solvents become less polar with increased temperature. For example, at elevated temperatures the bond angle in water widens and its dielectric properties approach those of organic solvents. Water at 250 °C actually has similar dielectric properties as acetonitrile at room temperature. Thus, water can be used as a pseudo-organic solvent at elevated temperatures where organic molecules will dissolve, not only because of the temperature, but also because of the change in dielectric properties. This makes some reactions that normally would not run in water possible.
- Solvents with low boiling points (e.g. methanol, dichloromethane and acetone), have lower reaction temperatures due to the pressure build-up in the vessel. If a higher absolute temperature is desirable to achieve a fast reaction it is advisable to change to a closely related solvent with a higher boiling point, dichloroethane instead of dichloromethane.

To show the responses of various solvents to microwave irradiation, we measured the temperature and pressure of pure solvents after 100 seconds of microwave irradiation. **Fixed Hold Time** was set to Off, **Absorption Level** was set to **Normal** and **Temperature** was set to 250 °C.

Solvent table

Solvent (Volume=2.5 mL)	Boiling Point (1 atm) (°C)	Attained Temp (°C)	Attained Pressure (bar)
1-Methyl-2-pyrrolidinone (NMP)	202	250	1
1,2-Dichloroethane	83	180	5
1,2-Dimethoxyethane (DME)*	85	130	3
1,4-Dioxane*	100	56	0
Acetone	56	150	7
Acetonitrile	81	180	13
Dichloromethane	40	110	5
Dimethylsulfoxide (DMSO)	189	250	1
Ethanol	78	155	13
Methanol	65	145	17
N,N-Dimethylformamide (DMF)	153	250	4
o-Dichlorobenzene	180	250	2
Tetrahydrofuran (THF)*	65	110	3
Water*	100	165	10
Xylenes*	137	50	0

** Some solvents can reach higher temperatures if they contain microwave-absorbing material and are heated for a longer time. For the less microwave-absorbing solvents, much higher temperatures have been observed in various reactions, for example: xylene (150 °C), 1,4-dioxane (200 °C), water (220 °C), 1,2-dimethoxyethane (200 °C), and tetrahydrofuran (180 °C).*

Getting started with microwave synthesis

Volume
Do not exceed or fall below the vial's specified volumes. Too low a volume will give an incorrect temperature measurement; while too high a volume does not leave sufficient head space for pressure build-up.

Biotage Microwave Vials 0.2-0.5 mL (only with EXP systems)

Biotage Microwave Vials 0.5-2 mL

Biotage Microwave Vials 2-5 mL

Biotage Microwave Vials 10-20 mL (only with EXP systems)

For more information on Biotage Microwave Vials, see www.biotage.com.

Concentration
The concentration depends on the type of chemistry that is performed. A unimolecular reaction is independent of concentration and can be performed in very dilute solutions. Bi- or tri-molecular reactions on the other hand are highly dependent on the concentration; a higher concentration gives a faster reaction. The maximum obtainable concentration is dependent on the properties of the substrates and reagents as well as the properties of the solvent(s) used.

Phase
All different phases can be used, i.e. solution phase, solid phase, solid supported reagents, solvent free and scavenger resins.

Stirring
Remember to always add a magnetic stirring bar to the microwave vial.

Inert atmosphere
In general inert atmosphere is not initially employed in microwave chemistry, and often not needed even if the reaction is carried out in this way conventionally. If needed, flush the vial with an inert gas before capping.

Time
Typically, most reactions require 2 - 15 minutes of irradiation.

Temperature
All synthesis systems from Biotage work in a temperature range between 60 °C and 250 °C. Optimally the used reaction temperature should be as high as substrates and products allow before they start decomposing or as high as the reaction solvent allows, whichever is lowest.

Pressure
The reactions can safely be performed at pressures of up to 20 bar. If the pressure in a vial becomes higher, the heating is automatically stopped and cooling begins. For an indication of the expected pressure of a reaction, please use the solvent table or the vapor pressure calculator at www.biotagepathfinder.com.

Time prediction

Most times, reactions proceed faster using microwave synthesis simply because they are conducted at higher temperatures. This chart provides an easy method to estimate the time it will take to run a reaction at a different temperature than reported. Based on the Arrhenius equation, it uses the coarse rule of thumb that a ten-degree increase in reaction temperature doubles the reaction speed. For example, if your reaction took **4 hours** at **140 °C**, it will take approximately **2 hours** at **150 °C** (see white numbers in the chart below).

Change in field color represents change in unit (hour/minutes/seconds). For example, if your reaction took **6 hours** at **100 °C**, it will take approximately **5 minutes** at **160 °C** (see red numbers in the chart below.)

Prediction chart

Temp (°C)	Times - change in field color represents change in unit									
20	1	2	4	6	8	12	24	48	96	172
30	30	1	2	3	4	6	12	24	48	86
40	15	30	1	1.5	2	3	6	12	24	43
50	8	15	30	45	1	1.5	3	6	12	22
60	4	8	15	23	30	45	1.5	3	6	11
70	2	4	8	11	15	23	45	1.5	3	5
80	56	2	4	6	8	11	23	45	1.5	3
90	28	56	2	3	4	6	11	23	45	1
100	14	28	56	1	2	3	6	11	23	40
110	7	14	28	42	56	1	3	6	11	20
120	4	7	14	21	28	42	1	3	6	10
130	2	4	7	11	14	21	42	1	3	5
140	53	2	4	5	7	11	21	42	1	3
150	26	53	2	3	4	5	11	21	42	1
160	13	26	53	1	2	3	5	11	21	38
170	7	13	26	40	53	1	3	5	11	19
180	3	7	13	20	26	40	1	3	5	9
190	2	3	7	10	13	20	40	1	3	5
200	1	2	3	5	7	10	20	40	1	2
210		1	2	2	3	5	10	20	40	1
220			1	1	2	2	5	10	20	35
230					1	1	2	5	10	18
240						1	1	2	5	9
250								1	2	4

With courtesy of David Rudge, AstraZeneca, Macclesfield, UK

Getting started with microwave synthesis

Optimize your reaction

Optimizing a microwave synthesis is very similar to optimizing a conventional synthesis. If your first reaction was not a success, changing the target temperature and reaction time can cause significant improvement. All remaining parameters that you would usually vary (i.e. concentration, solvent, reagent, etc.) should be varied when applicable.

If the reaction is not proceeding at all or not going to completion:

- Increase the temperature. As long as the reactants/reagents can withstand the higher temperature, the only limit is the pressure build-up in the vial and the security limit of 250 °C.
- Extend the reaction time.
- Increase the concentration(s) of reagent(s).
- Change the solvent. Some solvents, (e.g. water), will behave differently at high temperatures as they become less polar. This makes some reactions that normally would not work in polar solvents perfectly feasible.
- Change the reagent(s). Due to the high temperatures that can be reached, sometimes a less reactive, but more temperature stable, reagent can be used.

If you see decomposition of reactants/reagents/products:

- Lower the temperature.
- Shorten the reaction time. It may be that the desired product is actually formed, but then decomposes rapidly at elevated temperatures. In some cases it is therefore possible to "trap" the product by using a shorter reaction time.
- Decrease the concentration(s) of reagent(s).
- Change to a more temperature stable reagent.

Clean the microwave cavity and IR-sensor

Notice: Handle chemical and liquid waste according to the Material Safety Data Sheets and to local/national guidelines on laboratory safety procedures.

The microwave cavity and IR-sensor must be cleaned after the occurrence of a microwave vial breakage or leakage.

You need the T20 TORX® screwdriver supplied with the instrument, a vacuum cleaner, a soft lens cleaning tissue (or similar), cotton swabs, soft and clean cloths, an emery cloth, a waste tray insert, pressurized air, water and/or alcohol. The cleaning solution is dependent on the residues inside the cavity.

Note: If the cavity air guide and/or the cavity lid seal are broken or distorted, you have to replace them.

1. Shut down the system and disconnect the power cord:

I. If the instrument is processing and you need to clean the microwave cavity at once, press **Stop** to abort the task in progress. If the **Processing Stopped** dialog appears, follow the instructions provided in the dialog.

II. Shut down the system, i.e. press **Main Menu**/**Log Out** and then **Shut Down**. If the **Warning High Pressure and/or Temperature** dialog appears due to remaining high pressure and/or temperature inside the microwave vial, carefully follow the instructions provided in the dialog.

TORX is a registered trademark of Textron Inc.

III. When the system has shut down, switch off the system and disconnect the power cord.

2. Clean the microwave cavity and IR-sensor:

I. If the system is equipped with a robot, remove the rack(s).

II. Remove the cavity cover (A) by lifting it and then pulling it towards you.

III. If a cavity insert (B) and/or a microwave vial are located in the microwave cavity, remove them. Clean the cavity insert using a cloth.

IV. Remove the service lid (C) by undoing the four screws and disconnecting the air tubing (D). Push in the blue collect against the black fitting and pull the tubing out.

V. Remove the cavity lid seal (E) by undoing the screw and carefully pulling out the lid seal.

VI. Clean the cavity lid seal with water or alcohol containing mild soap. Do not use aromatic or chlorinated solvents.

Warning:

- Ensure that the equipment is switched off and the power cord is disconnected before cleaning the microwave cavity and IR-sensor.
- Do not attempt to operate the equipment if the microwave cavity contains trapped objects or moisture. There is a risk of damage to the equipment and microwave leakage.
- In the event of a microwave vial breakage inside the microwave cavity, the cavity and the waste tray may contain harmful residues and broken glass.
- Make sure that the cavity cover, waste tray, waste tray insert and waste lid are in position when the instrument is processing. If a microwave vial would break or leak inside the microwave cavity, and the cavity cover, waste tray, waste tray insert and waste lid are not in their positions, there is a risk of personal injury.
- When cleaning the microwave cavity, ensure that the lid seals are in place before reassembling the service lid. If a lid seal is removed or distorted, there is a risk of exposure to excessive microwave energy.

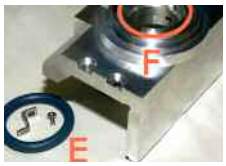
Maintenance

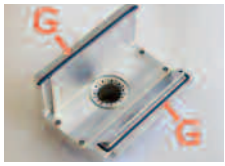
Note: If the lid seal is broken or distorted, it has to be replaced.

VII. Clean the service lid, using a cloth.

VIII. Clean the seal slot (F), using an emery cloth.

IX. Make sure that the service lid and all its parts are dry and that the two service lid seals (G), on the back of the service lid, are in place. If a seal is broken or distorted, replace it.



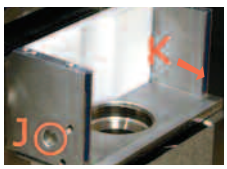


X. Put the cavity lid seal back in place.

Note: Do not tighten the screw too hard.

XI. Remove the IR-sensor (H) from the microwave cavity by undoing the screw.

XII. Clean the IR-sensor, using a soft lens cleaning tissue (or similar) dampened with distilled water, alcohol or Kodak lens cleaner. Do not scratch the surface! If the IR-sensor is broken, contact service at Biotage.



XIII. Remove the cavity air guide (I) and clean it using a cloth. If the cavity air guide is broken or distorted, replace it.

XIIV. If possible, vacuum the microwave cavity. Otherwise remove as much as possible of the spill with a soft and clean cloth.

XV. Clean the microwave cavity, including the IR-housing (J), using pressurized air, a cloth and cotton swabs.

XVI. Make sure that all parts are dry and that the two service lid seals (K), on the side of the cavity wall, are in place. If a seal is broken or distorted, replace it.

XVII. Reassemble the IR-sensor, cavity air guide, service lid, air tubing and cavity insert.

Note: Make sure to insert the cavity air guide correctly with the hole facing the IR-housing.

XVIII. If the inside of the cavity lid (L) needs to be cleaned, hold a cloth beneath the cavity lid while flushing with a solvent suitable for the residues.

3. Clean the waste tray and replace the waste tray insert:

I. Undo the waste tray screw (M) and unhook the waste tray (N).

II. Remove the waste lid (O) and empty the waste tray of the disposable waste tray insert (P) with collected glass and chemical debris in a designated waste container.

III. Clean the waste tray.

IV. Make sure that the waste tray is dry, and then insert a new waste tray insert into the waste tray and put the waste lid back in place.

V. Remount the waste tray on the cavity wall.

4. Put the cavity cover back in place and perform a reference run:

I. Put the cavity cover and, if the system is equipped with a robot, the rack(s) back in place.

II. Connect the power cord and switch on the system.

III. Check the IR-sensor and the pressure sensor by performing a reference run as described below.

Perform a reference run

A reference run should be performed when it is required to check the accuracy of the temperature and/or pressure readings, e.g. after the microwave cavity and IR-sensor has been cleaned due to a microwave vial breakage.

To perform a reference run:

1. Cap an unused microwave vial containing 5 mL of deionized water and a magnetic stirring bar.

2. Select the **Editor** tab (in the **Chemistry** mode) and set up a reaction with the process parameters 250 °C and 5 minutes.

3. Press **Run** and follow the instructions in the **Load Experiment** dialog.

4. Monitor the temperature versus pressure at the **Status** tab and check that the readings are within the limits listed below:

Temperature	Pressure
100 °C	0.4 ± 0.4 bar
160 °C	5.2 ± 1.7 bar
180 °C	9.0 ± 2.0 bar
200 °C	14.5 ± 2.5 bar
214 – 224 °C	22 bar

5. If the readings are not within the limits, the instrument needs to be calibrated. Contact 1-POINT SUPPORT™ at Biotage. Visit www.biotage.com for contact information.

For more information on how to set up an experiment, see the online help.

Release remaining pressure manually

Only release remaining pressure inside the microwave vial manually by pressing the **Vent** button in the **Warning High Pressure and/or Temperature** dialog that appears due to remaining high pressure and/or temperature inside the microwave vial. Carefully follow the instructions appearing on the screen.

Clean the exterior of the instrument

If the touch screen has been contaminated by chemicals, it must be cleaned immediately.

To clean the exterior of the instrument:

1. Shut down the system, i.e. press **Main Menu**/**Log Out** and then **Shut Down**.

2. When the system has shut down, switch off the system and disconnect the power cord.

3. Clean the touch screen and the exterior of the instrument, using a soft and clean cloth. The cloth can be dry or dampened with neutral detergent or alcohol.

4. When you are done cleaning the instrument, connect the power cord and switch on the system.

Warning: Ensure that the equipment is switched off and the power cord is disconnected before cleaning the exterior of the instrument.

General information

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Information in this document is subject to change without notice and does not represent any commitment from Biotage AB.

User documentation

All user documentation available for Initiator™ can be downloaded at www.biotage.com.

Consumables and accessories

In order to maintain compliance, only consumables and accessories supplied by Biotage must be used in the equipment. To order consumables and accessories, visit our website www.biotage.com.

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You have access to our in-house team of organic chemists with extensive experience of microwave synthesis, and medicinal and combinatorial chemistry. We provide online chemistry support and keep you up to date with the latest advances in the field.


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