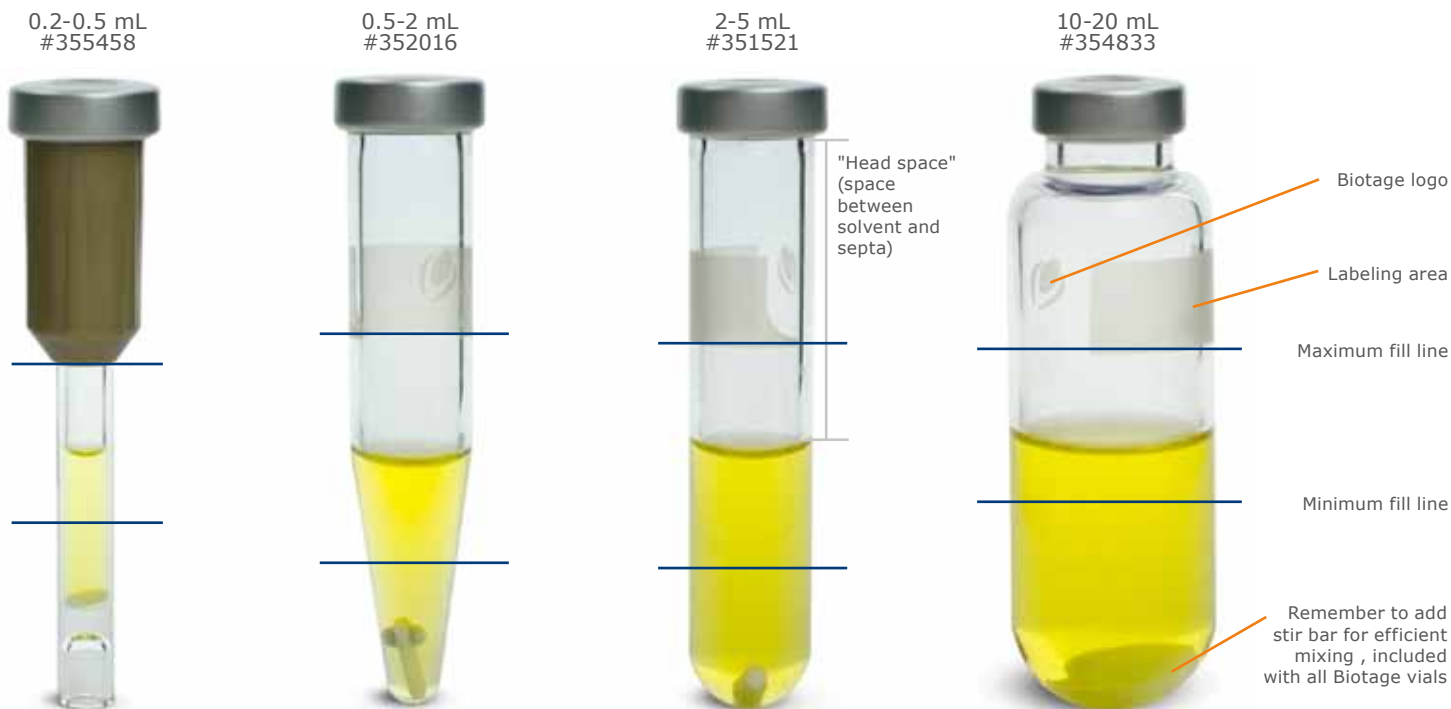


Success with Microwave Synthesis

Quick Guide to Initiator+

Set Up Your Reaction Vial

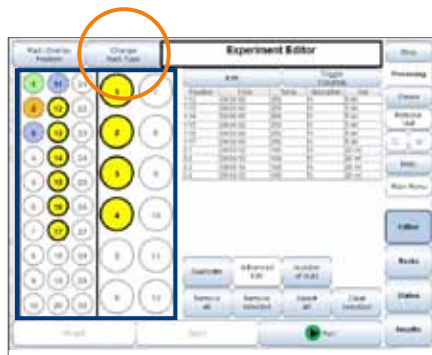
Shown actual size, simply hold your prepared reaction vial up to the photo to check for proper filling. All Biotage microwave synthesizers are calibrated for temperature and pressure using Biotage vials.



Intuitive Touch-Logic Control™

1. Set Up Synthesizer for Your Reaction

For systems equipped with Robot 8 or Robot 60, press the **Change Rack Type** key. For all other systems proceed to step 2. Select the vial positions to load. With the vial(s) highlighted proceed to the **Edit** tab.



2. Optimize Synthesizer Conditions and Start Run

Method parameters are listed at left, edit as desired, be sure to press **ENTER** after each change. Specify the reaction time, temperature and pre-stirring³ time. Next, select vial size, absorption² level and fixed hold time³. Press **Apply**. View each vial's parameters and proceed to **Run**.



3. Monitor the Status

Check reaction status and remaining processing time. View the temperature, pressure and power. The **Edit Current Reaction** tab allows reaction parameters to be altered while the reaction is still being processed.



¹For heterogenous, biphasic and exothermic mixtures. ²Normal=Organic solvents/substrates; High/V.High=Ionic content. ³ON=Time counts down after set temperature is achieved.

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Notes for Success

If you expect: One of the substrates is gaseous (NH₃, NH₄OH) at high temperatures.
Solution: Start at temperatures lower than 2 times the boiling point of the solvent and increase the head space

If you expect: Performing high temperature reaction in solvents that develop high pressures (eg. MeOH, EtOH).
Solution: If you don't know the pressure profile of a solvent, start at a maximum temperature equal to 2 times the boiling point of the solvent.

If you expect: Reactions produce rapid and high amounts of gaseous byproduct.
Solution: Provide enough head space by using the lowest volume possible (see diagram for proper filling). Start your testing with low concentration of the reaction mixture (e.g. about 0.1 M)

Expect: Reactions are extremely exothermic.
Solution: Start using low temperatures with absorption level setting at *Very High*. Dilute reaction mixture if possible.

Solvent Table

Solvent	Boiling point (°C)	Temp (°C)	Pressure* (bar)
N,N-Dimethylformamide (DMF)	153	250	5
Water	100	220	16
Ethanol	78	180	16
Methanol	65	160	17
N-Methylpyrrolidinone (NMP)	202	220	5
Ammonium hydroxide (28%)	—	150	19
Dimethylsulfoxide (DMSO)	189	250	5
Pyridine	115	220	8
1,2-Dimethoxyethane (DME)	85	200	6
Dichloromethane (DCM)	40	140	15
Acetonitrile	86	200	10
O-Dichlorobenzene	190	250	2
1,4-Dioxane	101	200	4
Acetone	56	150	5
Tetrahydrofuran (THF)	65-67	180	12
Xylene	137-138	150	2
1,2-Dichloroethane (DCE)	83	170	2
Toluene	110	170	4
Diethyl ether	35	135	4

This is a general table and this data was generated under various reaction conditions. The actual temperature and pressure achieved with a given solvent might differ significantly with different reaction conditions and compositions. Blue indicates "good" microwave absorption, while gray indicates "poor" microwave absorption.

* Volume = 2.5 mL

Vial preparation:

1) Remember to add stir bar for efficient mixing. Catalysts, salts, or visible precipitate should be washed clear of head space and into solution. Particles adhering to glass head space could cause excessive heating increasing possibility of failure of vial. Stay within specified vial volume range (see diagram for proper filling).

2) The vial on the right is capped properly. It should not be possible to turn the cap by hand. The top of the cap should be flat. The vial to the left is capped too tightly.



Time Prediction – Conversion from Conventional to Microwave Heated Synthesis

To transfer a method from conventional to microwave heating, simply use the Initiator wizard.

1) Input the time in minutes from your conventional heating process, touch **NEXT >>**.

2) Input the temperature at which the conventional reaction was performed, touch **NEXT >>**.

3) Increase the temperature step wise until the time is between 5 and 15 minutes. Touch **NEXT >>**, Initiator then loads these reaction conditions and is ready to run.

This wizard can then be run over and over again to fine tune the settings or you can tweak each individual setting by re-using your previous reaction conditions.



Learn more about synthetic procedures – sign up for **Biotage® Pathfinder** today at www.biotagepathfinder.com and enjoy a rich library of reactions.

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