



Academic Solutions

Microwave Synthesis Systems and Teaching Materials



Undergraduate Instruction

Graduate Research

Advanced Microwave Technology

We Simplify Science



Transform your undergraduate organic laboratory into a state-of-the-art learning experience.



Real World Education

Expose your students to the most modern methods and the complex chemistries that they will be using after graduation, including multi-step and multi-component reactions that you currently cannot teach due to time restrictions. Microwave energy has become the method of choice for chemists, as it offers the safest, most effective way to increase reaction rates and improve product yields, while promoting green chemistry. The Discover® 2.0 and MARS 6™ Synthesis microwave synthesizers from CEM provide users with the safety, flexibility, and reproducibility demanded by teaching and research laboratories no matter the size of the group.

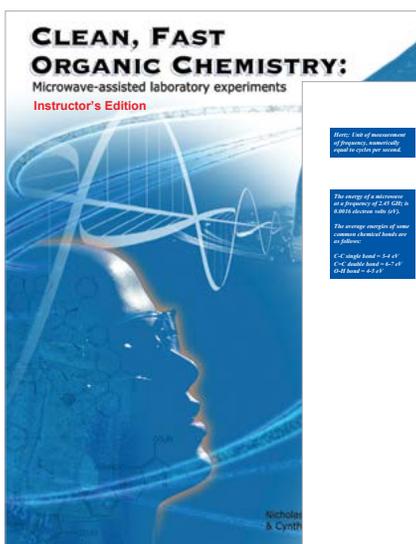
Literature

Clean, Fast Organic Chemistry: Microwave-assisted laboratory experiments

Not sure how to integrate a microwave into your existing class? The authors of Clean, Fast Organic Chemistry have taken many standard organic chemistry experiments and converted them to utilize microwave assisted heating techniques and green chemistry principles.

Experiment Type	Reflux Conditions	Microwave Conditions
Diels-Alder 	90 minutes in DMF	10 minutes in H ₂ O
Williamson Ether Synthesis	60 minutes in H ₂ O	10 minutes in H ₂ O
Aldol Condensation	23 hours in H ₂ O	15 minutes in H ₂ O
Bromination	45 minutes in HOAc	8 minutes in HOAc
Nucleophilic Aromatic Substitution 	60-90 mins in Toluene	10 minutes in EtOH / H ₂ O
Hydrolysis	34 hours in MeOH / H ₂ O	9-15 minutes in MeOH / H ₂ O

 = Green chemistry experiment



Microwave Energy

Microwave irradiation is a form of energy that falls between 300 and 300,000 megahertz (MHz), relatively low on the electromagnetic spectrum (Figure 1). Unlike ultraviolet radiation, which is used in photochemistry and can break chemical bonds, microwaves are low frequency forms of energy that cause molecules to rotate.

The energy of a microwave is a frequency of 2.45 GHz in 8000 decibels (dB).

The energy of some common chemical bonds are as follows:

- C-C single bond = 3 eV
- C=C double bond = 6.7 eV
- C≡C triple bond = 10 eV

Like all electromagnetic energy, microwaves move at the speed of light and are composed of oscillating electric and magnetic fields. The electric field and magnetic field are perpendicular to each other and perpendicular to the direction of travel. It is primarily the electric field of the microwaves that causes the rotation of molecules and causes the transfer of energy and the generation of heat.

Figure 1. The electromagnetic spectrum

Figure 2. A microwave

Microwave heating is dependent on the polarizability of the molecules. Polar molecules are heated more rapidly than non-polar molecules. The energy is added at a rate faster than the molecules are able to relax, all of the molecules in solution will be in a constant state of disequilibrium, providing more than enough energy to overcome the activation energy barrier (E_a) and drive the reaction to completion (Figure 7).

With microwave irradiation, since the energy is interacting with the molecules at a very fast rate, the molecules do not have time to relax and the heat generated can be, for short times, much greater than the overall rounded temperature of the bulk reaction mixture. In essence, there will be instantaneous localized superheating (Figure 5).

Figure 5. Localized superheating of molecules in solutions

The rate of a reaction is described by the Arrhenius equation, which expresses the relationship between the rate of reaction and the activation energy, E_a (Figure 6).

Figure 6. Arrhenius equation

The activation energy is the energy barrier that must be overcome in order for the reaction to occur. A microwave transfers energy to the reaction every nanosecond (10^{-9} seconds). The almost constant energy input is achieved at a rate greater than the molecular relaxation rate, which is on the order of 10^7 seconds. Because the energy is added at a rate faster than the molecules are able to relax, all of the molecules in solution will be in a constant state of disequilibrium, providing more than enough energy to overcome the activation energy barrier (E_a) and drive the reaction to completion (Figure 7).

The Arrhenius equation is a simple, yet very accurate predictor of the reaction rate.

$$k = Ae^{-E_a/RT}$$

k = rate constant
 A = pre-exponential factor
 E_a = activation energy
 R = gas constant
 T = absolute temperature

Lab Safety

Not only is microwave-assisted chemistry good for the environment, it is also safer for chemists. Microwave synthesis systems designed for the laboratory offer an unmatched level of safety.

- Eliminate hot plate burns
- Reactions return to room temperature before removing from microwave
- Reactions are completely contained
- Laboratory grade microwave systems provide monitoring and feedback control of temperature, pressure, and stirring to ensure maximum safety and reproducibility in the lab.

Go Green

It's time to think about the environment and our impact on it. Microwave energy is an inherently efficient way to transfer energy to a reaction, as it is transferred directly to the reaction solution. Because of this quality, it is the ideal energy source for driving reactions.

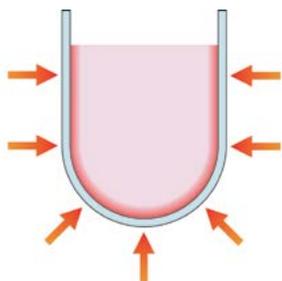
- Use water, ethanol or other environmentally benign solvents
- Neat reactions/high conversions help eliminate waste
- Non-hazardous reagents help students design safer syntheses
- Use catalysts, not stoichiometric reagents



Microwave Heating 101

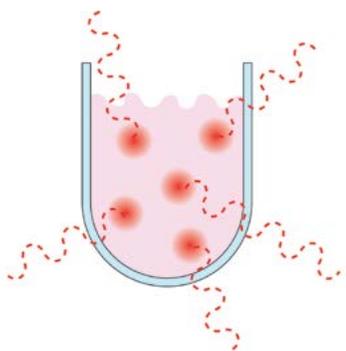
Convective Heating

Reactions typically covered in academic organic laboratories rely on convective heating, a method in which energy is transferred indirectly to the reactants by applying heat to the outside surface of the vessel. This form of heating is very slow and inefficient which is why many experiments require lengthy reflux times and leave little time for characterization, purification or repetition.



Microwave Heating

With microwave heating, the vessel wall becomes virtually transparent to microwaves allowing energy to be directly absorbed by the reaction and provide instantaneous activation or localized super-heating of the molecules in solution. This direct molecular activation limits side reactions and provides a fast and efficient form of heating. Reactions that previously took hours, or even days, to complete can be performed in minutes.



Microwave Solutions for Your Lab

CEM offers two microwave platforms for synthetic chemistry: the Discover 2.0 and the MARS 6 Synthesis. The Discover 2.0 is a sequential system, which is best suited for a smaller class size and split research/teaching needs. It can also be utilized in a larger lab section where students work in groups and coupled with an auto-sampler for high-throughput labs. The MARS 6 is a parallel reactor designed for batch processing of reactions. The ability to run multiple reaction vessels simultaneously is advantageous for large teaching laboratories, as it only takes 30 minutes to complete a set of up to 36 vessels.

Discover 2.0

Ideal for classes of 12 students or less

The Discover 2.0 is a single mode, sequential microwave system which allows significant flexibility in the teaching lab, as students can explore different reaction parameters and work with various substrates and solvents. The sequential format is ideal for classes of 12 students or less, but also fits in classes with a larger number of students who work in groups.

The Discover is also the system of choice for research laboratories performing initial investigative syntheses and chemistry optimization with a variety of accessories available, including automation decks.



MARS 6 Synthesis

Ideal for classes of 13 students or more

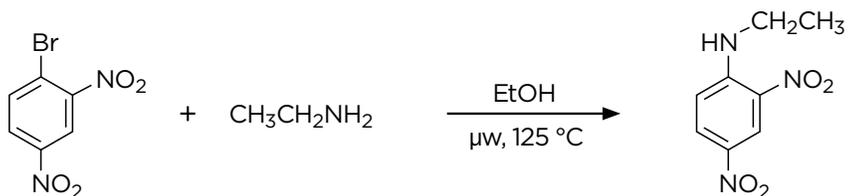
The best-selling MARS 6 is a multi-mode microwave system that provides parallel reaction processing, making it ideally suited for teaching laboratories. With the ability to accommodate up to 36 pressurized vessels or up to a 5 L open flask, the MARS 6 offers both high-throughput for larger lab sections and flexibility to run batch syntheses.

In addition to accommodating synthetic chemistry, the MARS 6 can also be used for solvent extraction and acid digestion for metals analysis.



Nucleophilic Aromatic Substitution

Complete in 10 minutes or less in the microwave.



	Discover 2.0	MARS 6 Synthesis
Maximum Reaction Vessels	1	36
Solvent: Ethanol	3 mL	5 mL
Reagent: 1-Bromo-2,4-dinitrobenzene	0.298 g	0.298 g
Reagent: Ethylamine (70% aqueous)	0.380 mL	0.380 mL
Reaction Time	6 minutes	10 minutes
Cool-down Time	2 minutes	20 minutes

Leadbeater, N. E.; McGowan, C. B. *Clean, Fast Organic Chemistry: Microwave-assisted laboratory experiments*. 2006, CEM Publishing.





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United States (Headquarters)

800-726-3331
704-821-7015
info@cem.com

France

33 (01) 69 35 57 80
info.fr@cem.com

Germany, Austria, Switzerland

(49) 2842-9644-0
info@cem.de

Ireland

+353 (0) 1 885 1752
info.ireland@cem.com

Italy

(+39)035896224
info.srl@cem.com

Japan

+81-3-5793-8542
info@cemjapan.co.jp

United Kingdom

(44) 1280-822873
info.uk@cem.com

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