GEN1 – Introduction to Microwave Heating

Estimated Time Required: 30 minutes

Introduction

A microwave is a form of energy found at the low energy end of the electromagnetic spectrum, with wavelengths between one meter and one millimeter (Figure 1). Just as the molecules that compose leftover food can be heated by microwaves, so can the molecules of a reaction in a chemistry lab.

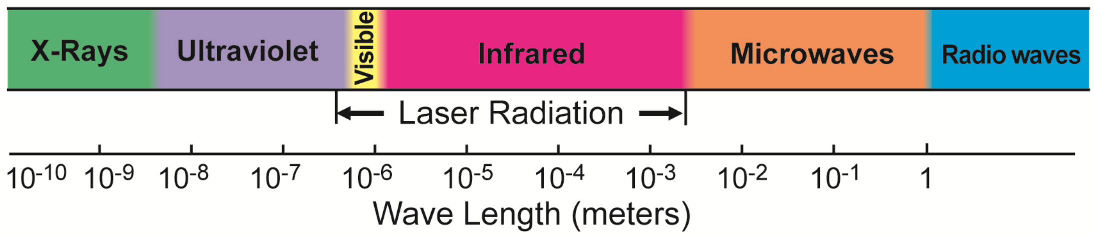

Figure 1. The electromagnetic spectrum.

As with any type of electromagnetic energy, microwaves are composed of perpendicular, oscillating electric and magnetic fields. It is primarily the electric field of a microwave that interacts with molecules, doing so through two methods: dipole rotation and ionic conduction. In Figure 2, a water molecule is shown interacting with an electric field. A dipole is shown on the water molecule. This dipole is due to: the difference in electronegativity values of oxygen and hydrogen, combined with the structure of the molecule, resulting in an electron gradient. With the oscillation of the electric field of the microwave, the water molecule rotates back and forth constantly trying to align its dipole with the changing field. Friction and heat result from this movement, termed dipole rotation.

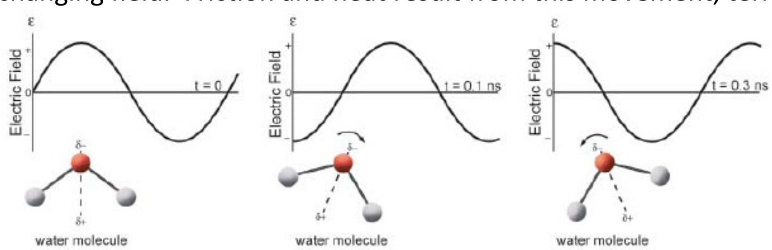

Figure 2. Methods of microwave heating: dipole rotation.

If there are free ions or ionic species in the reaction mixture, then microwave energy can be transferred via ionic conduction. Figure 3 shows a chlorine ion interacting with the same electric field. The charged atom tries to align itself with the field, moving back and forth. The movement of ions in the electric field generates heat, which also affects the transfer of energy. The higher the temperature of the reaction mixture, the more efficient the transfer of energy becomes.

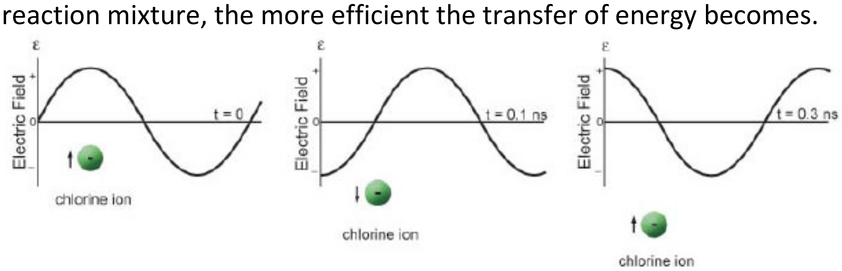

Figure 3. Methods of microwave heating: ionic conduction.

Because they interact directly with the molecules of a reaction mixture, microwaves transfer energy more rapidly and efficiently than conventional techniques that rely on thermal conductivity, where heat is transferred to the vessel first and then to the solution. The following procedure will explore dipole rotation and ionic conduction through a series of experiments.

Experimental

Reagent	CAS	MW (g/mol)	Density (g/mL)	mmol	Amount
deionized water				2.75	
brine (saturated sodium chloride solution)					2.00 mL
hexanes (a mixture of isomers)				18.3 or 9.16	
ethanol				34.3	
iron (II,III) oxide				0.432	
benzaldehyde				9.84	
trihexyltetradecylphosphonium chloride				1.72	

Required equipment: Discover® microwave synthesizer with Synergy software
Microsoft® Excel® or comparable data graphing program
10-mL glass microwave reaction vessels (7)
small stir bar (7)
microwave vessel cap (11)

Ramp to Temperature

1) Pipet 2.00 mL of deionized water into a 10-mL microwave reaction vial with a stir bar.
2) Cap the vial and place into the microwave cavity.
3) Program a "Dynamic" method, heating the solution to 150 °C using 300 W for a 1 minute ramp period, then hold at this temperature for 30 seconds.
NOTE: If the Synergy software is not being used, create a method on the Discover using "Standard" control.
4) For Synergy data collection, skip to step 5. For manual data collection, record the temperature and power every 15 seconds and graph data versus time. .
5) Once the reaction vessel has cooled to <50 °C, open and empty the contents into the appropriate waste container (retaining the stir bar with a magnet).
6) Repeat step **1)** thru **5)**, using 2.00 mL of brine.
7) Repeat step **1)** thru **5)**, using 2.00 mL of hexanes.
8) Repeat step **1)** thru **5)**, using 2.00 mL of ethanol.
9) Repeat step **1)** thru **5)**, using a mixture of Fe_3O_4 (0.100 g) and hexanes (2.00 mL).
10) Repeat step **1)** thru **5)**, using a solution of benzaldehyde (1.00 mL) and hexanes (1.00 mL). **Ensure that the solution is homogeneous before starting the method.**
11) Repeat step **1)** thru **5)**, using a solution of an ionic liquid (0.50 mL) and hexanes (1.00 mL). **Ensure that the solution is homogeneous before starting the method.**
12) Graph the temperature and power data.
   a. Using the Synergy software, right click on the data file and select "Save to file", saving the files as a .csv format and graph.
   b. Using manual data collection, enter the temperature and power data into a .csv file and graph.

Fixed Power

1) Pipet 2.00 mL of ethanol into a 10-mL microwave reaction vial with a stir bar.
2) Cap the vial and place into the microwave cavity.
3) Program a "Fixed Power" method, heating the solution to 150 °C using 300 W for 30 seconds.
4) For manual data collection, record the temperature and power every 15 seconds and graph data versus time. For Synergy data collection, skip to step 5.
5) Once the reaction vessel has cooled to <50 °C, open and empty the contents into the appropriate waste container (retaining the stir bar with a magnet).
6) Repeat steps **1)** thru **5),** using 2mL Hexanes.
7) Repeat steps **1)** thru **5),** using a solution of benzaldehyde (1.00 mL) and hexanes (2.00 mL). **Ensure that the solution is homogeneous before starting the method.**
8) Repeat steps **1)** thru **5),** using a mixture of Fe_3O_4 (0.200 g) and hexanes (2.00 mL). **Ensure that the solution is homogeneous before starting the method.**
9) Graph the temperature data for all 4 samples on the same chart.
   - For automatic data collection, export the data and graph the temperature and power data in a CSV file.
   - For manual data collection, enter the temperature and power numbers into a CSV file and graph.

Results and Discussion

1) Compare the following graphs for the "ramp to temperature" experiments, commenting on which sample heats better and requires less power. Why? What is the method of microwave heating in each example?
   a. Temperature and power: water vs. brine
   b. Temperature and power: hexanes vs. ethanol
   c. Temperature and power: hexanes vs. Fe_3O_4/hexanes
   d. Temperature and power: hexanes vs. benzaldehyde/hexanes
   e. Temperature and power: hexanes vs. ionic liquid/hexanes

2) Compare the 4 graphs for the "fixed power" method all on the same plot. Can you make a **true** statement about which molecule better absorbs microwave energy? Why or why not?