



Online Chemistry Course (OLCC)

Chemical Safety: Protecting Ourselves And Our Environment

Paper: 2.b

Discussion: September 12 – 18, 2004

Title: *Safety Issues Related to Microwave-Enhanced Chemistry*

Author: Barbara L. Foster

Affiliation: West Virginia University, Morgantown WV

Posted: 10/29/2004

Website: http://science.widener.edu/svb/olcc_safety/index.html

SAFETY ISSUES RELATED TO MICROWAVE-ENHANCED CHEMISTRY

Barbara L. Foster
C. Eugene Bennett Department of Chemistry
Eberly College of Arts and Sciences
West Virginia University
Morgantown, WV 26506-6045
(304) 293-2729
bfoster@wvu.edu

INTRODUCTION

Why do chemists use microwave ovens in their laboratories?

Chemistry was defined in Dr. Johnson's Dictionary of 1775 as "an art whereby sensible bodies contained in vessels...are so changed by means of certain instruments, and principally fire, that their several powers and virtues are thereby discovered, with a view to philosophy or medicine."¹ Thankfully, nowadays, we do not use "fire" in the strictest sense to heat chemicals in the laboratory to obtain our desired results.

Scientists use various methods to stimulate chemical reactions that would otherwise not take place under ambient conditions. Many researchers have discovered that the process of microwave heating of chemicals can be more energy efficient than conventional heating methods, such as oil baths, steam baths, heating mantles, and hot plates, which use convection and conduction principles. When liquids or solids are heated in a microwave oven, the microwaves pass through the transparent reaction vessel and are absorbed by the reaction mixture, thus transforming electromagnetic energy into heat, which drives the chemical reaction. Accelerated reaction rates and higher yields are desired results that can be achieved by using microwaves. When using microwaves to heat reactions, less time is required to achieve the desired result (i.e., oxidations, esterifications, hydrolyses, Diels-Alder reactions, ether preparations). However, these same characteristics often make the reactions more difficult to control and reproduce.

At home, consumers regularly use microwave ovens to warm snacks or cook meals because they are quick and convenient to use, but domestic-grade microwave ovens should never be used to heat chemicals in a laboratory. They do not possess the inherent safety features that are necessary to avoid accidents in the laboratory. To avoid worker injury, costly fires, explosions, and facilities damage, researchers should purchase laboratory-grade microwave ovens with built-in safety features for their experimental work involving the heating of chemicals.

SAFETY FEATURES IN LABORATORY-GRADE MICROWAVE OVENS

What standard safety features should be included in a laboratory-grade microwave oven to protect the laboratory worker?

When purchasing a laboratory-grade microwave oven, one should ensure that the oven includes the following safety features:

- A ventilation system that protects the laboratory worker by preventing exposure to flammable and toxic materials.
- A solvent sensor that detects the presence of significant amounts of flammable organic solvents in the microwave cavity and can be designed to automatically shut down the microwave before the lower explosive limit (LEL) concentration in air is attained. Solvent sensors can also be located in the exhaust system.
- An electric interlock system that is designed to prevent the operation of the instrument in the event of fan failure or obstruction of the venting system.
- A feedback control system that utilizes a temperature probe to prevent overheating of samples by controlling the temperature of the solution based on reaction profiles and standard methods.
- A rotator and wave stirrer to minimize temperature variations within the cavity.
- An adjustable-speed air pump that can produce even distribution of temperature.
- A shorter cycle time selector to provide more precise temperature control.
- The complete isolation of the reaction cavity from all electronic components to prevent corrosion of control and safety circuits and to prevent electrical ignition of solvents. The most common sources of ignition in the laboratory are gas flames and heating elements.
- A pressure release mechanism for laboratory microwave vessels that is designed to protect the unit and the laboratory workers in the event of a complete failure of the vessel.
- The cavity should be grounded, unable to float electrically, and coated with a non-conducting material (i.e., Teflon) to minimize the potential of high-energy discharge.
- The microwave oven door should be able to withstand an event equivalent to the explosion of 1g TNT.

These built-in safety features generally translate into higher list prices for laboratory-grade equipment. The typical list price for a 1200W, 1.6 cu.ft. domestic-grade microwave oven is approximately \$120. The typical list price for a laboratory-grade microwave oven that is comparable in size can begin at \$5000.²

APPLICABLE CODES

Are there any federal codes or standards that apply to the use of microwave ovens in the laboratory?

The National Fire Protection Association (NFPA) “NFPA 45 - Standard on Fire Protection for Laboratories Using Chemicals, 2000 Edition” was designed “to control hazards and protect personnel from the toxic, corrosive, or other harmful effects of chemicals to which personnel might be exposed as a result of fire or explosion”(1.2.2)³ The stated purpose of NFPA 45 is “to provide basic requirements for the protection of life and property through prevention and control of fires and explosions involving the use of chemicals in laboratory-scale operations” (1.2.1) . Section 9.1.2.1 (Heating Operations) states, “All heating of flammable or combustible liquids shall be conducted so as to minimize fire hazards.” The standard advises that flammable or combustible vapors that evolve during drying operations should be vented to avoid ignition (Section 9.1.6 (Other Operations). Section 9.2.3.4 (Heating Equipment) states “Laboratory heating equipment such as ovens, furnaces, environmental chambers, and other heated enclosures shall not be used to heat, store, or test flammable or combustible liquids or aerosols containing flammable gases unless the equipment is designed or modified to prevent internal explosion.”

Microwave ovens that are not explosion-proof quality do not conform to NFPA 45 and are considered to be ignition sources, along with other less obvious electrical sources such as refrigerators, stirrer motors, and heat-guns. Most standard operating procedures for microwave ovens and Material Safety Data Sheets (MSDS) for flammable liquids clearly state that flammable liquids should not be heated up or produced in microwave ovens. The OSHA Laboratory Standard, 29 CFR 1910.1450, “Occupational Exposure to Hazardous Chemicals in Laboratories”, defines a flammable liquid as a “liquid having a flash point below 100°F (37.8°C), except any mixture having components with flash points of 100°F (37.8°C) or higher, the total of which make up 99 percent or more of the total volume of the mixture.”⁴ The Laboratory Standard defines an explosive chemical as “a chemical that causes a sudden, almost instantaneous release of pressure, gas, and heat when subjected to sudden shock, pressure, or high temperature.”

According to the August 8, 2002 OSHA Standard Interpretation, “Applicable OSHA standards and safety considerations for microwave device use in a laboratory”, possible hazards that should be considered when using a microwave oven in a laboratory setting include:

- Microwave radiation may leak from damaged door seals, hinges, or latch, or because someone defeats the interlock system.
- When flammable or combustible materials are heated and the temperature nears the flash point, fire or explosion is more likely. (The superheated state is unstable and it can very rapidly convert to a gaseous state at the boiling point, retaining a substantial quantity of vapor. If the flash point of the liquid is reached, ignition may occur.)

- When flammable liquids are heated in a closed area, the vapor concentration increases and may exceed the lower flammable limit.
- The electrical wiring can provide an ignition source.
- The electrical wiring could be damaged or not installed correctly.
- When hazardous chemicals are heated in a closed device, employees will be exposed to higher than usual vapor concentrations when the device is opened.
- The microwave radiation or the heating process may act on chemicals to produce more hazardous chemical by-products.⁵

Additionally, synthetic reactions in which elemental metals are the resulting product may cause electrical arcing, a sustained luminous electric discharge across a gap, which occurs at high temperatures.⁶ The resulting arcing becomes an excellent ignition source and can damage the oven, which can lead to microwave leakage.

An accident which occurred at an environmental laboratory involved the rapid evolution of a gas from a microwave digestion vessel during the microwave digestion of two dissimilar samples of unknown composition. The microwave oven door was blown off of its hinges and was transformed into a missile, which landed approximately 15 feet from its origin, striking a lab bench during its sojourn. It should be noted that similar accidents have occurred in the United States in an analytical laboratory at a Fortune 500 company, a chemical plant laboratory, a pharmaceutical laboratory, a medical laboratory, and a government laboratory.⁷

GREEN CHEMISTRY

How do the principles of Green Chemistry apply to microwave-enhanced chemistry?

According to the U.S. Environmental Protection Agency “12 Principles of Green Chemistry”, the Twelfth Principle states: *Minimize the potential for accidents: Design chemicals and their forms (solid, liquid, or gas) to minimize the potential for chemical accidents including explosions, fires, and releases to the environment.*⁸ Although some researchers believe that the concepts of Green Chemistry can be applied to microwave heating techniques, these concepts do not apply when using flammable liquids in microwave ovens. The use of flammable liquids in domestic-grade microwave ovens has resulted in explosions and fires in the laboratory.⁹

Using a domestic-grade microwave oven to conduct experimental work involving flammable liquids violates the Twelfth Principle. An energy savings benefit does not justify the potential for injury and property loss when using a domestic-grade microwave oven.

CONCLUSIONS

The emerging field of microwave-enhanced chemistry is exciting and full of promise for the researcher. Microwave ovens allow the chemist to produce higher yields of the desired product in less time. Experimental work that involves heating flammable liquids should never be performed in a domestic-grade microwave oven, which lacks the safety features that are necessary to avoid injuries, explosions, and fires. To avoid injuries and property loss, researchers must purchase and utilize laboratory-grade microwave ovens that possess requisite safety features.

REFERENCES

- ¹ D. Michael P. Mingos, D.R. Baghurst. Microwave Enhanced Chemistry: Fundamentals, Sample Preparation, and Applications, H. M. Kingston, Editor, Chapter 1: “Applications of Microwave Dielectric Heating Effects to Synthetic Problems in Chemistry”, American Chemical Society: Washington, D.C., 1997.
- ² M.E. Cournoyer, B.L. Foster. “The Use of Microwave Ovens with Flammable Liquids”, 2003.
- ³ National Fire Protection Association, NFPA 45: Standard on Fire Protection for Laboratories Using Chemicals; Quincy, MA, 2000 edition.
- ⁴ Occupational Safety and Health Administration, *Occupational Exposure to Hazardous Chemicals in Laboratories*; 29 CFR; Part 1910.1450, 1990.
- ⁵ U.S. Department of Labor, OSHA Standard Interpretation, Response to Dr. Gary Login, 08/08/2002, “Applicable OSHA standards and safety considerations for microwave device use in a laboratory.”
- ⁶ Rieke, Rueben D.; Sell, Matthew S.; Klein, Walter R.; Chen, Tian-an; Brown, Jeffrey D.; Hanson, Mark V. “Rieke metals: highly reactive metal powders prepared by alkali metal reduction of metal salts,” *Active Metals* (1996), 1-59.
- ⁷ H. M. (Skip) Kingston, P.J. Walter, W.G. Englehart, and P.J. Parsons, Laboratory Microwave Safety, Chapter 16: Microwave Enhanced Chemistry: Fundamentals, Sample Preparation, and Applications, Chapter 16 “Microwave Laboratory Safety”, American Chemical Society: Washington, D.C., 1997, <http://www.sampleprep.duq.edu/dir/mwavechap16/mwave.htm#appendixc>
- ⁸ U.S. Environmental Protection Agency Green Chemistry Principles, <http://www.epa.gov/greenchemistry/principles.html> (accessed October 2004)
- ⁹ D.M.P. Mingos, D.R. Baghurst, *Chem. Soc. Rev.* 1991, 20.1; C.R. Strauss, R.W. Trainor, *Austr. J. Chem.* 1995, 48, 1665.