Microwave Instruments: Green Machines for Green Chemistry?

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Microwave Instruments: Green Machines for Green Chemistry?

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It's a fair assumption that almost all of your students use a microwave oven regularly. But how many of your students know that microwaves can be easily adapted for laboratory use? Integrating a microwave system into laboratory teaching provides an entry into synthesis, spectroscopy, and analytical methods while allowing for the introduction of the ethics and the philosophy inherent in green chemistry. Because of the ubiquity of the microwave oven in today's society, students will readily accept its use as a laboratory tool and integrate its use into their own knowledge bank.

Applications of Microwave Systems in the Undergraduate Laboratory

We learned of microwave methods through being introduced to green chemistry. Green chemistry is an exciting field to explore because of the possibility of reducing the amounts of chlorinated solvents, wastewater, and energy consumption in teaching and research. These are practices we hope to instill in our students. Microwave ovens also provide a way to introduce to the laboratory green-chemistry methods such as solvent-free syntheses and atom economy, two examples of the 12 principles of green chemistry *(1)*. Adoption of microwave methods in the teaching lab can also provide benefits such as reduced sample processing time and improved extraction and digestion efficiencies for other applications.

As the number of applications of microwave technology in the research laboratory has grown, the use of microwave methods in the undergraduate laboratory has also expanded. In this *Journal* and in other venues, most articles dealing with microwave systems have an organic chemistry focus. Microwave-assisted organic synthesis (MAOS) is now quite common and has been used in the undergraduate laboratory for the synthesis of aspirin *(1)*, Wittig reactions *(2)*, Knoevenagel condensations *(3)*, Diels–Alder reactions, Williamson ether synthesis *(4)*, as well as for hydrolysis, oxidation, and esterification *(5)*. Microwave methods have even been applied to a one-semester organic course in which Miller and Leadbeater developed a microwave preparation of biodiesel from vegetable oil *(6)*. In that activity, the authors were looking to demonstrate the relevancy of chemistry to engineering students.

The use of microwave systems can also be readily applied in teaching undergraduate inorganic chemistry and analytical chemistry. For example, in the inorganic chemistry laboratory, we have had students use a microwave oven to synthesize mesotetraphenylporphyrin without the use of solvents *(7)*. After dissolving a metal salt into a solvent such as methanol, we use the microwave oven to insert a variety of metals into the porphyrin ligands. Having been trained for years not to place metals into domestic microwave ovens, it was delightful to learn that as long as the metal was dissolved, it could be safely placed into the oven.

Although we have not seen the application of microwave instrumentation to analytical chemistry published in teaching journals, many analytical chemists have put microwave instruments to use in microwave-assisted extraction (MAE). For example, a solvent-free microwave method has been developed for extracting oils from herbs *(8)*. Environmental applications include the extraction of pesticides from plants, and companies provide application notes to assist in digesting samples such as impatiens and geraniums. We envision microwave methods readily adapted for use in every undergraduate laboratory, and look forward to using them.

Choices, Choices: What Do You Need? What's Available?

Once you have embraced the idea of using a microwave instrument in an undergraduate laboratory, this question logically follows: what type of microwave system should you buy?

When we first started exploring the use of microwaves in the laboratory, some colleagues suggested using an inexpensive domestic model. After all, a domestic microwave oven can cost less than $100, and many organic chemists have brought such microwave ovens into the undergraduate laboratory *(1, 2)*. We were never quite comfortable with this, however, and therefore obtained (through successful grant proposals) two laboratory-grade microwave instruments to use in undergraduate teaching. One microwave system has been used to perform organic synthesis reactions in an undergraduate research lab; a second system was integrated into both the organic and inorganic chemistry laboratory curricula. After two years of use, these endeavors have proved to be well worth our effort.

Laboratory-grade microwave instruments are distinctly different from your kitchen microwave oven! Domestic microwave ovens are incompatible with many laboratory solvents and do not offer programmed temperature, pressure, and power control. Domestic microwave ovens are also not safely vented, and the simultaneous processing of multiple samples can be awkward (at best). Although kitchen microwave ovens can function in the teaching lab for the quick and routine heating of non-noxious aqueous solutions, they are unsafe for complicated synthetic, extraction, or digestion procedures. Laboratory-grade microwave instruments are much safer. Safety features such as proper ventilation, vapor sensing, and pressure relief on vessels allow for greater control of the experimental environment. A 220 V power line is required, and safe venting requires that you feed a vent hose from the microwave into a hood unless the microwave is already in the hood.

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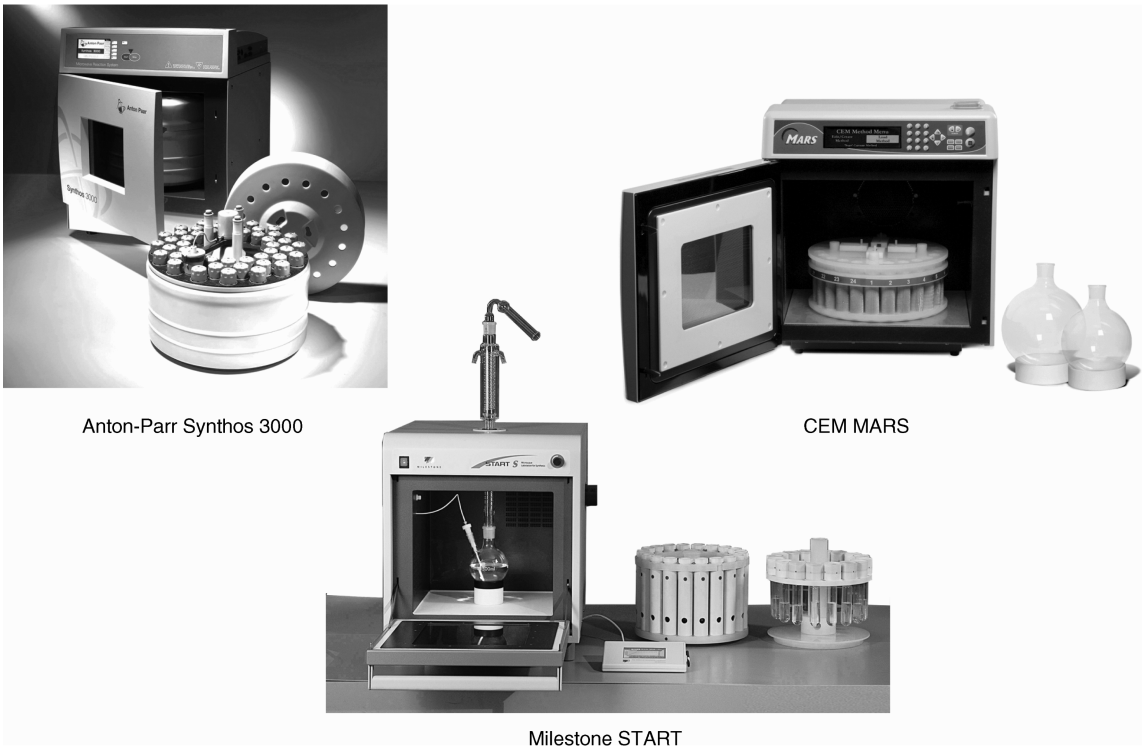

Figure 1. Anton-Parr Synthos 3000 (top left), CEM MARS (top right), and Milestone START (bottom), are all laboratory-grade microwave systems commonly used for undergraduate teaching. The photographs show rotor options that allow an entire class to complete a reaction simultaneously. Photos are used with permission.

Depending on your budget and the anticipated uses, several microwave options are available. Before purchasing a particular instrument, you will need to consider your likely applications. Will the instrument be used for synthesis, extraction, or digestion procedures, or perhaps for all three? What numbers and volumes of samples will you need to process during one lab session? The answers to these questions will dictate the vessel material best suited for your needs: typically glass, quartz, or polytetrafluoroethylene. A variety of rotor types are also available. The rotors securely hold the pressurized vessels and rotate different sizes and numbers of vessels. For use in the academic laboratory, rotors accommodating up to 24 student reactions are available. Vessels can break or corrode over time, and replacement vessels can be costly, so purchasing a few extra vessels up front is a good investment. The laboratory-grade microwave instruments always measure temperature and power; options are also available to measure pressure. Depending on your needs and options, a typical teaching laboratory microwave instrument will cost between \$20,000–30,000. But do not forget to ask for educational discounts!

Although we purchased CEM microwave systems *(9)*, we also carefully considered Milestone products *(10)*. CEM and Milestone are two major players in a field of companies entirely devoted to laboratory microwave systems. Anton-Paar *(11)* is another supplier that carries microwave instruments as part of a larger instrument suite (Figure 1). Of special interest to educators is that all three companies provide plenty of support and Web-based resources to help you get started quickly. Milestone and CEM also provide hardcopy resources: Milestone offers *Clean Chemistry: Techniques for the Modern Laboratory (12)*, and CEM publishes the laboratory manual *Clean Fast Organic Chemistry: Microwave-Assisted Laboratory Experiments (13)*.

Setup and Usage of Microwave Instruments

Initially we were concerned about how quickly the microwave would be ready for laboratory use. Would the instrument's installation and operation be challenging to put to use with 16 or more students? Fortunately, the system's setup was quite simple; in the organic laboratory, we were running experiments in a few days. Using the microwave is not like training for using an NMR or a GC–MS; the learning curve is not steep. Moreover, no hidden upkeep or maintenance issues have emerged. We use the microwave when appropriate, and then turn the instrument off and walk away until its next use. Because people are so accustomed to using microwave ovens at home, its use in the laboratory is simply an extension of an everyday tool.

We hope that more undergraduate educators investigate the use of microwave systems in the curriculum. While the laboratory-grade systems are expensive in comparison to domestic ovens, we have found the former easy to use and adapt. Once you show others in your department how easy microwave instruments are to work with, we predict that your peers will use them as well. We encourage you to examine journals such as *Green Chemistry Letters and Reviews (14)* and *Green Chemistry (15)* as well as microwave instrument manufacturer Web sites for more ideas on how to bring microwave systems into your teaching laboratory.

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