Microscale Chemistry in the USA

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Abstract

Microscale Chemistry has become the method of choice for the teaching laboratories in the United States. This introspective look at the development of microscale Chemistry details the advantages promised by microscale Chemistry during the early developmental phase of microscale in the United States. These advantages are then compared to the current usage of microscale. This comparison is designed to highlight areas for potential development of the field.

Resumen

La química en microescala se ha convertido en el método seleccionado para la enseñanza de laboratorios en los Estados Unidos. Esta mirada introspectiva al desarrollo de la química en microescala detalla las ventajas prometidas por la Química en Microescala durante la fase de desarrollo inicial en los Estados Unidos. Estas ventajas se comparan entonces con la utilización actual de la microescala. Esta comparación trata de subrayar las áreas potenciales para el desarrollo de este campo.

The advent of the microscale revolution in the United States is legitimately traced to the pioneering book by Dana Mayo, Ronald Pike, and Samuel Butcher (Mayo *et al.*, 1986) hereafter referred to as MPB. Interestingly, the MPB approach was not the first microscale approach marketed in the United States. A kit designed by Horak and Morris was marketed briefly by the Arthur H. Thomas Company in the mid-70's. Since this approach did not achieve commercial success, the start of the microscale revolution must be attributed to a unique combination of timing, materials, and personnel. Regulations requiring a minimization of the amount of hazardous material produced at any facility in the USA (Resource Conservation and Recovery Act (RCRA, 1976)) and regulations concerning the disposal of hazardous waste (Toxic Substance Control Act, (TSCA, 1976)) had been passed by the American Congress (Pine, 1990). Forward thinking people began to advocate the adoption of this approach. Commercially, Albert Klein and John Ryan of Ace Glass, Inc. in Vineland, NJ, began sales of the glassware advocated by MPB. In academic laboratories, Anthony Winston at West Virginia University brought the microscale approach to an advanced laboratory course, even before the textbook had been published. In fact, in the first year of adoption for this advanced laboratory course, a Xerox copy of the draft version of the MPB textbook in a loose-leaf notebook was used as the textbook for the course. The first workshop on the use of microscale glassware in the organic Chemistry laboratory at West Virginia University occurred in 1985 with Ronald Pike, Anthony Winston, and this author presiding over the conference. Within a short period of time, several other laboratory textbooks in the general area of organic Chemistry appeared, some of which adhered to the glassware and techniques of the MPB approach (Pavia *et al.*, 1990; Moore and Winston, 1996) while others advocated another glassware approach and different experiments (Williamson, 1987; Holman, 1991). From this start in organic Chemistry, the adoption of microscale as the standard for laboratory technique took off at a fast pace and is now estimated to be used at 75-80% of the academic organic Chemistry laboratories.

Outside of the area of organic Chemistry, a number of authors were busy introducing microscale books and techniques into the high school scene (Ehrenkranz, 1991; Ehrenkranz and Mauch, 1993; Russo, 1995; Mills, 1991). At a slightly later time, microscale Chemistry has even further moved into all areas of University-taught Chemistry, with textbooks appearing in general Chemistry (Szafran *et al.*, 1993; Szafran *et al.*, 1992; Singh *et al.*, 1995; Williamson and Little, 1997) and inorganic Chemistry (Szafran *et al.*, 1991). The large number of books appearing on this subject make it tempting to say that microscale techniques can be considered to be the laboratory standard, at least for this generation of students.

Since this author has been involved throughout the chronology of microscale development, an introspective look at microscale through the eyes of this

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organic chemist may help to point towards areas for future developments in this field.

What is microscale Chemistry?

An excellent discussion of the use of the word "microscale" in contemporary context is found in the preface of the MPB book (Mayo *et al.*, 1986). In this preface, a clear chronology of the miniaturization of the organic laboratory is presented. Starting with the 4th edition of the textbook by Levy which was published in 1902 (Levy, 1902), and which used approximately 50 g of starting material in a standard preparation, the scale of a laboratory experiment has been gradually converted to a quantity involving ≤ 100 milligrams of the MPB approach. Since that time, the term microscale has been used to indicate different quantities, depending upon the perspective of the textbook authors. In the area of organic Chemistry, most authors have settled into a range, wherein the term microscale refers to 50-200 mg of solid materials or solution volumes ranging from 0-5 mL of total volume. Just to show how different audiences perceive this term differently, qualitative analysis schemes in high school laboratory experiments use 1-2 drops of reagent to observe color changes or the formation of precipitate (Ehrenkranz and Mauch, 1993; Russo, 1995). In fact, a commercially available Chemistry set designed for students aged 10-18 by the Smithsonian Institution utilizes microscale amounts of 1-2 drops for certain experiments. The unifying principal throughout all of these approaches is that the quantities of starting materials have been dramatically reduced from the quantities used in previous times.

What was the driving force that propelled us into microscale Chemistry?

In order to look at the development of microscale Chemistry and assess the current status of microscale, a reexamination of the originally stated goals of the microscale laboratory is useful. The microscale approach was said to offer both economical and pedagogical advantages, when compared to the formerly used macroscale approach. Normally, the pedagogical advantages were stressed when persuading faculty colleagues of the need to convert the laboratories to the microscale approach. In contrast, the economic advantages were stressed when persuading University administrators to spend the necessary funds for major glassware purchases and other equipment required for the retooling of the lab. A more detailed look at these advantages is given below.

Pedagogical advantages of microscale Chemistry

In 1985, the major pedagogical advantages of the microscale approach were argued to be safety, better student technique, time savings in the laboratory, and new reaction possibilities. Many of the advantages have now been documented, as detailed below.

Safety

Significant advantages in safety for the students arise from the introduction of microscale techniques into the teaching laboratory. The major safety advantage of the microscale approach is the exposure of the students to smaller quantities of hazardous materials. This advantage arises from the fact that there are smaller quantities of chemicals to be adsorbed through the skin and vapors from volatile solvents are present in smaller quantity. In fact, this advantage has been utilized to defer the construction or retrofitting of teaching laboratories with expensive air-handling equipment. A further safety advantage arises from the hazards due to explosion and/or fire in the teaching laboratory. The ultimate experiment utilizing this advantage is the published procedure for the microscale synthesis of 2,4,6-trinitrotoluene (TNT) (Russel, 1990). Although this author is unaware of any laboratories that have adopted this procedure as a regular component of their Program, the scale of the experiment does make this reaction to be an interesting possibility.

Better student technique

The handling of small amounts of material requires the students to pay more attention to detail in order to have a successful experience in the laboratory. Although those inclined to oppose the introduction of microscale techniques into their laboratory have seen this point as a disadvantage of the microscale approach, laboratory results conclusively demonstrate that microscale does, indeed, result in better technique.

In one example of a laboratory demonstration of the better student results originating in the WVU organic Chemistry laboratory, a laboratory section of 64 students achieved a 62% yield during a Craig tube crystallization of acetanilide in their initial crystallization experiment, while those using the traditional Hirsch or Büchner funnel techniques achieved only

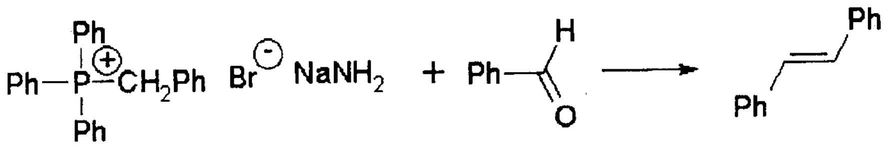


Figure 1. Synthesis of *trans*-stilbene.

a 53% yield for the same experiment. Similar results detailing better yields have been obtained in other laboratories (Pavia *et al.*, 1990).

Time savings

The miniaturization of the amounts of chemicals used in an experiment leads to a miniaturization of the size of the glassware required to perform a given laboratory operation. Since this miniaturized equipment can most often be held in one hand, the assembly of a set of glassware to run an experiment is dramatically diminished in most organic laboratory settings. Disassembly also takes less time for the same reasons. Qualitative experiments in which only drops of reagent are added to determine the products of the reaction (*e.g.*, a colored precipitate) now take only seconds.

The time savings produced from this miniaturization leads to two significant advantages in the teaching laboratory. First, when students make mistakes (as some certainly will), they can begin the experiment anew, without a substantial time penalty. This is in dramatic contrast to previously used macroscale techniques in which the options for the instructor included only sending the student home, giving the student a poor grade, or both. The second time savings advantage is that the reduced time for completing an experiment could be used to develop critical thinking skills in the students. The enhancement in the critical thinking skills could be manifested through a variety of techniques (*e.g.*, more detailed questions, enhanced interpretation of experimental results, more experiments).

New reaction possibilities

The conversion to the microscale laboratories brings with it a cost savings (see below). Mayo, Pike and Butcher (Mayo *et al.*, 1996) have amply demonstrated that experiments with "expensive" reagents could now be utilized in the curriculum. For example, MPB use the "instant-ylid" (*i.e.*, benzyltriphenylphosphonium bromide/sodium amide) for the synthesis of trans-stilbene as shown in the equation below (Figure 1).

The instant-ylid normally costs $4-5/gram, making this reaction unthinkable for a normal macroscale laboratory experiment, which might require a cost of $20/student/experiment for this reagent alone. However, when each student needs only 600 mg, the cost/student of ca. $2/student/experiment makes the experiment feasible.

Economic advantages of microscale Chemistry

The use of smaller amounts of material leads naturally to a decrease in the operating costs of a teaching laboratory. The savings derive from the areas of acquisition, waste disposal, and legal liability.

Acquisition costs

The savings are estimated simply by considering the quantity of chemicals used in the microscale approach when compared to the macroscale approach. If the scale of a reaction is cut from ca. 1 g to 100 mg, then the cost of a microscale reaction will cost approximately 10% of the corresponding macroscale reaction (*i.e.*, a 90% cost savings). Since the use of solvents for the reaction and for glassware cleaning do not scale exactly to these proportions, a more realistic cost savings of 80% is conservatively calculated when comparing the microscale approach to the macroscale approach.

Waste disposal costs

Although the waste disposal costs are generally not considered as part of the economic picture in any laboratory setting, these hidden costs may contribute more to the operation of a lab than the acquisition costs of the chemicals. During our efforts to introduce microscale Chemistry to the organic laboratories of WVU, we were able to demonstrate that the purchase cost of all chemicals and solvents for the organic laboratories was ca. $5,000, while the cost for disposing of waste materials for these same laboratories was ca. $10,000 (Collins, 1985). At first glance, these numbers seem out of line. However, they are a true reflection of the short-term and long-term concerns of the hazardous waste haulers (see below). Waste disposal cost figures for today's laboratory are unavailable, but are estimated to be a minimum of three to five times larger than the acquisition costs.

Legal liability

Depending on the laws of the country (or local government), the legal liability for hazardous waste disposal can be enormous. In the United States, the hazardous waste disposal laws (TSCA) dictate that individuals or corporations can be sued to recoup monies from placement of materials in hazardous waste sites, even if the material was placed in the site prior to the enactment of the federal regulations. The "cradle-to-grave" responsibility, as mandated by the legislation, was intended to ensure that University officials (or officials from any organization) do not look in the other direction when hazardous waste is transported away from the institution's boundaries. Whether or not these governmental actions are just, the facts are that several institutions have been required to face legal action with potential fines of $200,000 which arose from concerns over chemical liability. Exact details of this type of activity are difficult to obtain, since offending institutions usually invoke secrecy agreements about settlements in hazardous waste settlements. Nonetheless, these laws raise issues that cannot be ignored by a responsible administration.

Payback time for purchase of equipment

As detailed above, the cost savings from using the microscale approach are significant. Although the purchase price of the equipment is expensive at first glance, the savings from using the microscale approach are sufficient to repay the purchase costs in a short period of time. For example, the glassware and other equipment needed for the miniaturization of the organic teaching laboratory at WVU cost ca. $30,000.

However, when one considers the yearly costs that West Virginia University incurred when conducting the organic laboratory ($5,000 for chemical acquisition and $10,000 for disposal), the conservative figure of 80% cost savings indicates that $12,000/year would be saved by converting to the microscale approach. This yearly savings repaid the initial investment of $30,000 in two years. After that time, the total amount of money spent by the University to provide chemicals for the laboratory was significantly decreased relative to the original chemical costs. There are few administrators who would not push for such a long term decrease in operating costs for the institution when these cost savings could be used elsewhere in the institution.

CURRENT ASSESSMENT OF THE STATUS OF MICROSCALE CHEMISTRY IN THE UNITED STATES IN 1998

Penetration of microscale techniques into the chemical curriculum

Exact details of the percentage of schools that now use microscale techniques are difficult to obtain, since there are no National survey instruments that collect this data. However, a more global perspective may be obtained from sales personnel (either textbook or glassware) who visit many different institutions than from a single University faculty member who sits at his/her own University and thinks that the entire universe is viewable from that perspective. Based upon conversations with a large number of sales personnel, this author estimates that ca. 75-80% of the available organic Chemistry laboratories in the US have converted to the microscale techniques.

One of the more interesting observations about the development of microscale is that the high school Chemistry community was the second group of chemists to adopt microscale techniques. With an army of textbooks, and enthusiasm that this change in curriculum was better for student learning, a large number of high schools have changed to microscale techniques as the standard technique. In fact, the Chemistry Olympiad in the United States has utilized microscale techniques as the standard technique.

Interestingly, the general Chemistry course at the College/University level in the United States has been the slowest unit to convert to microscale. Several factors enter into this phenomenon. Since most of the Chemistry is aqueous solvent-based, and the products of the reactions are non-hazardous materials (*e.g.*, NaCl), the ability to pour "waste" solutions down the drain is an ever-present factor in determining whether waste is generated in any reaction. The perception that little chemical waste is generated has been one factor which has slowed the development of textbooks. Another factor which has slowed the rate of adoption of microscale in general Chemistry is that the general Chemistry curriculum is usually taught by several individuals at larger universities. Leadership and direction of the course is then run by a committee. Since committee decisions are always more difficult than individual decisions, the easiest committee decision is to not change from their current curriculum.

Comparison of 1985 argued advantages to the practice of today

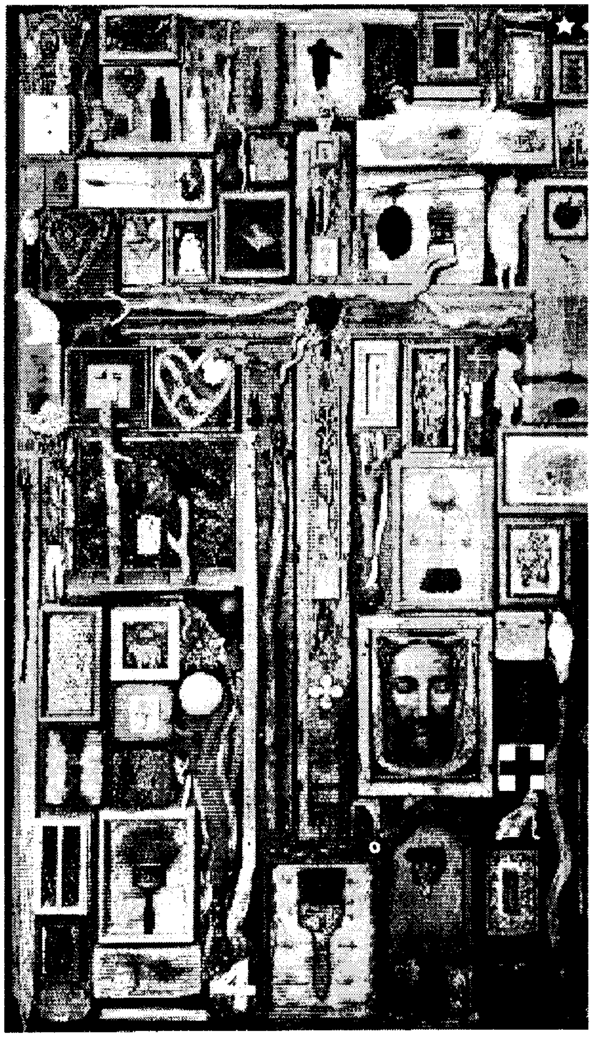
While the arguments for conversion to the microscale approach were, and still are, persuasive, it is instructive to reevaluate each of the proposed advantages of microscale as to whether the full potential of the microscale approach has been achieved.

Economic advantages of microscale Chemistry

In all three areas (*i.e.*, acquisition costs, waste disposal costs, and potential legal liability), the microscale approach performs well. Since the total amount of chemicals used and the production of waste has diminished, the overall direct costs have diminished as promised in 1985. Only in the area of legal liability does microscale still show any potential weakness, since legal liability is, by definition, omnipresent. This weakness is not really a weakness, since the decrease in chemical usage can only be viewed as being better than the previously used macroscale approach. The single path to do better with the issue of legal liability would be to completely eliminate the laboratory Program from the curriculum. While University administrators continue to revisit the idea of elimination of the laboratory from the curriculum, this author believes firmly that the laboratory experience is essential in teaching Chemistry effectively to students. The microscale approach allows for a cost effective way to continue the laboratory experience to a maximum number of students at a minimum cost.

Pedagogical advantages of microscale Chemistry

While the economic advantages of microscale have come to maximum fruition, the pedagogical advantages have not developed as extensively, at least in the area of organic Chemistry where the author is most familiar. Student technique has improved overall. However, there is still room for improvement. A common problem in the WVU laboratories is that students learn very early in the course how to perform the technique of crystallization and achieve higher yields than with the macroscale techniques. Later in the year, they forget this technique when they begin running reactions in the laboratory. A common mistake is to gain a 95% crude yield of product and then report an overall yield of 15% for the reaction when their yield from a recrystallization is only 20%. Vigilance and reinforcement of important techniques seems to be required to allow students to reach their full potential.



David McManaway, *Jomo Board #12*, 1984. Tomado de *The Sciences*, marzo-abril de 1999, p. 40.

In this author's opinion, the time savings gained from using microscale techniques has been underutilized. In the area of organic Chemistry, only the textbook of Moore and Winston (Moore and Winston, 1996) attempts to take advantage of the time savings by incorporating a more thorough Program of qualitative experiments designed to show various kinetic phenomena and reactivity patterns. Other approaches have utilized the large number of experiment offerings as additional experiments to fill the increased time available, leaving the adopters of the textbooks to decide upon additional experiments. To date, no use of the time to enhance critical thinking skills has been intentionally introduced into the laboratory Program.

Table 1. Professional Goals (Chem 133, Fall 1996).

	%
M.D.	29.22
Pharmacist	22.37
P.T.	9.13
Chemical Engineer	7.31
Other	7.31
Undecided	6.39
Veterinary	5.02
Biologist	4.11
Chemist	2.74
Dentistry	2.74
Environ.	0.91
Nutrition	0.91
Wildlife Man	0.91
Biochemist	0.46
Chiropractic	0.46
Teaching	0.00
Law	0.00

The area of safety has also been better, but there is still room for improvement. Although microscale has indeed proven to be more safe for the greater majority of students, individual students have found ingenious ways in which to injure themselves by new techniques. Therefore, vigilance and common sense must still be practiced to eliminate accidents completely from the laboratory.

Finally, the possibility to develop new reaction types has received only limited attention in the area of organic Chemistry. While there has been an explosion of new reaction types and compounds for preparation in the area of inorganic Chemistry, organic textbooks have tended to rely upon the well-tested recipes which have been performed on the macroscale level for many years. There is room for development of new experiments here.

Future of microscale Chemistry in the US and in the world

Microscale Chemistry is the future of laboratory teaching. Therefore, anything concerning the future of laboratories is something that affects the future of microscale Chemistry.

The following statements are designed to stimulate the next round of developments in the area of microscale. Although some of the areas for development have already been identified as areas in which microscale has not reached its full potential, other issues remain as improvements to microscale and the laboratory experience in general.

Relevance to student audience

Laboratory textbooks should relate to the student audience. Without relevance to their personal situations, students have less incentive to learn the desired material. In many Academic institutions, the trend of the organic Chemistry laboratory is to serve as a service course for those students entering the health profession. Although this author would not attempt to claim that the enrollment of students in his class was representative of every institution in the United States, the enrollment data are representative of trends in the entire country. With this qualification in mind, the professional goals of the students in the author's class for the fall 1996 semester are shown in Table1. Most interesting in this data is the percentage of students who are interested in becoming chemists (*i.e.*, 2.74%). The majority of the students (ca. 90%) are entering pre-health fields. Refusal of the next generation of textbooks to recognize this fact will only help to further drive the wedge between the public and the universities about the relevance of their teaching to the masses.

Environmental friendliness

Microscale Chemistry has been advanced in recent years as being "Green Chemistry", *i.e.*, environmentally friendly Chemistry. These arguments are better made by Szafran in another article in this Journal (see pages 102-106). Further improvements in this area could come through the use of written instructions on waste disposal procedures specifically incorporated into the experiments. The materials produced can be used for further procedures, thus minimizing the amount of material purchased and needing disposal at the end of the course. Further environmental friendliness could be obtained by using procedures that utilize naturally occurring compounds.

International movement(s)

The trend to microscale has passed an irreversible phase. No longer will anyone be going back to the old macroscale techniques. Active microscale movements are in place in several countries throughout the world. An attempt to indicate all groups that are struggling with the conversions to microscale Chemistry in the world would only lead to an embarrassing omission. It is sufficient to state that movements are currently found in all continents of the world.

Importance of computers

The dramatic revolution taking place in information technology (as evidenced by the computer) ensures that a conflict between a laboratory experience and the capabilities of computers will occur at some future point in time. As the capabilities of computer hardware and software increase, the potential capabilities of the computer to simulate the laboratory experience will become ever more realistic. Already, work has begun in some academic institutions to develop home laboratory procedures (*i.e.*, those that can be accomplished by distance learning). The innovators of the laboratory curriculum need to keep this issue in perspective as they develop the next generation of materials.

Summary

In conclusion, the microscale approach has been adopted by the vast majority of academic laboratories in the US. Ideas for future improvements in the area of microscale have been advanced for potential adoption by future textbook authors.

Acknowledgments

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