

A Major Revolution in the Chemistry Laboratory

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Nueva sección: Química en Microescala

En los últimos 10-15 años ha surgido un movimiento en muchos lugares del mundo para usar la Química en Microescala en los laboratorios de enseñanza.

De una manera sencilla, la Química en Microescala consiste en hacer más con menos. Sus ventajas, posibilidades y retos han sido discutidas y presentadas por muchas personas en varios foros y publicaciones. Este movimiento coincide con la generación de una conciencia ambiental que lleva a cuestionar los métodos tradicionales de vivir y de hacer las cosas, a favor de posibilidades más acordes con una cultura de conciencia ambiental y de racionalidad en la utilización de los recursos naturales.

Educación Química ha sido un vehículo de actualización en diversos aspectos de la enseñanza de la Química. Es por ello que ahora iniciamos una nueva sección en donde se pretende incluir nuevas técnicas, experimentos, discusiones que posibiliten la diseminación de la Química en Microescala para el beneficio de nuestros lectores.

Iniciaremos con una serie de artículos generados de las presentaciones realizadas por diferentes autores durante el Simposio: Microscale Chemistry, co-organizado por la Canadian Chemical Society (Dr. David Berry), la American Chemical Society (Dr. Mono M. Singh) y la Sociedad Química de México (Dr. Jorge Ibáñez Cornejo) y realizado en el V Congreso de América del Norte en Cancún, México, en noviembre de 1997.

La sección, pues, queda abierta. ¡Esperamos sus contribuciones!

Cordialmente,

Dr. Jorge Ibáñez Cornejo

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Editor de Sección

Abstract

A historical perspective on the origins of Microscale Chemistry in the USA and of the motivation behind it is presented here by one of the fathers of Microscale Chemistry and his colleagues. The success achieved can be understood in the light of the establishment of many programs and centers around the world.

Resumen

Se presenta un relato histórico del inicio de la Química en Microescala en los Estados Unidos y de los motivos que le dieron origen, escrito por uno de los padres de la Química en Microescala y sus colegas. El éxito alcanzado se vislumbra en el establecimiento de programas y centros alrededor del mundo.

Something had to be done!! But what? The time—1980 at Bowdoin College. The problem to solve? The organic laboratory situation. The laboratory was old, having extremely poor ventilation facilities and thus poor air quality. Certainly it was not in compliance with present EPA and OSHA regulations. Students were experiencing headaches and often nausea from the chemical fumes. Clothes smelled, fires often occurred when large amounts of ether were used and secretaries would leave the building on “Grignard” day! All these problems were due impart from the huge amount of solvents being used in the large scale (macroscale) experiments being performed. Complaints by staff and especially by students to the President of the College brought the situation to a head. Finally, a cost study was made to retrofit the laboratory—\$300,000—money that the College did not have.

The organic professors, Dana W. Mayo (Bowdoin) and Ronald M. Pike (Merrimack College—on Sabbatical leave at Bowdoin), and Samuel S. Butcher (Chair of the Chemistry Department, Bowdoin) debated the situation. The answer to the question—Isn't there a better way?—gave birth to the new idea of applying “microscale techniques” to the introductory organic laboratory! The revolution was about to start!!

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Questions immediately arose: Could the beginning students work with 50-150 mg quantities? Could experiments be developed at this scale? Did we have the incite to modify existing equipment and/or develop new miniature apparatus to accomplish the tasks? How could the techniques best be taught? Would going "micro" actually solve the air quality problem?—and on and on. There were some doubts; but there was a strong conviction that it was worth a try. It would involve a lot of work; no doubt about that. Should we write a laboratory manual? Would any one be willing to publish such a text?

We did go ahead and the result is history. Going "micro" was the right answer. Although it was a simple idea, it came at the right time. Trial laboratory sections in organic were conducted at Merrimack and Bowdoin in 1983. The initial results were extremely promising. Szafran, Pike and Singh also found this to be true when the concept was implemented in the inorganic area at Merrimack College (1986). It was found that the students could rapidly adapt to the microscale approach and, if anything, laboratory technique and performance improved markedly. The psychological impact was also beneficial, since there was a strong preference for working in a safer, more healthful environment. Today we often sit and contemplate what has taken place over the last eighteen years in regard to introducing the microscale concept (termed Small-scale Chemistry by IUPAC) into our instructional laboratories. One word describes it—awesome!! Not only is it being used extensively in the organic area, but the concept has also penetrated the inorganic field, the general Chemistry area, high school level instruction, and in several cases the elementary area.¹ The number of institutions involved in microscale instruction to students at all levels is extensive.

Mayo and Pike ran summer workshops in Microscale-Organic Chemistry at Bowdoin College from 1986-1992. Williamson at Mt. Holyoke College, and Penn and Winston at West Virginia University also offered workshops several years later. Pike, Szafran and Singh established the National Mi-

croscala Chemistry Center (NMC²) at Merrimack College, North Andover, MA in 1993. Its main focus: a training site for faculty and industrial personnel through the offering of an ongoing series of workshops at the elementary, high school, community college and college/university level. Areas of instruction presently include organic, inorganic, general, advanced general, industrial, high school and AP Chemistry. Instruction is also offered in chemical science for elementary school teachers. To further the worldwide dissemination of microscale Chemistry technology, NMC² has been instrumental in establishing additional microscale centers. Centers are currently in place at Roosevelt University (USA), West Virginia University (USA), Roxbury Community College (USA), Rhode Island Community College (USA), Pinkerton Academy (USA), Kokkolan Teknillinen Oppilaitos (Finland), Universidad Iberoamericana and Universidad Autónoma de San Luis Potosí (México), Deakin University (Australia), Halmstad High School (Sweden), and Universiteit van Amsterdam (The Netherlands).² The establishment of Centers in Puerto Rico and India are in the planning stage. This past year PETE (Partnership for Environmental Technology Education) has provided money for NMC² in conjunction with the CSMATE Program at Colorado State University (directed by S. Thompson), to establish Microscale Centers in the USA at six Community Colleges.³ Funds have recently become available for additional Centers in 1998-1999.

Historically, microscale techniques had been available for many years. The feasibility of carrying out Chemistry experiments at microscale levels was established in the mid 1800's, principally in central Europe with the work of Emish and Pregl.⁴ Pregl

¹ (a) M.M. Singh; D.A. Rigos, *Microscale Workbook for Elementary Science Teachers*, National Microscale Chemistry Center, Cussing Hall, Merrimack College, No. Andover, MA, 1995; (b) M.M. Singh; D.A. Rigos, *Työkirja Peruskoulun Kemianopettajille*, Nordic Microscale Chemistry Center, Kokkolan Teknillinen Oppilaitos, Kokkola, Finland.

² Addresses, telephone and in some cases Fax numbers for these Centers can be found on our Web page for NMC²: <http://www.silvertech.com/microscale>.

³ They are located at: State University of New York at Morrisville, NY; St. Johns River Community College, Palatka, FL; Kirkwood Community College, Cedar Rapids, IA; Tyler Community College, Tyler TX; Oregon Institute of Technology, Klamath Falls, OR; Santa Rosa Community College, Santa Rosa, CA.

⁴ For a paper on the "Evolution of Microscale Equipment" and a listing of other workers in the field at that time see: R. Hathaway in "Safety Considerations in the Microscale Laboratories", Symposium at the 197th National Meeting-ACS, Dallas, TX, April 1989, American Chemical Society, Washington, DC, 20036, 1991.

received the Nobel Prize for his microscale analytical work in 1923. In the United States, semimicroscale and microscale Chemistry gained a foothold shortly after World War II. Cheronis and Ma at Brooklyn College, Benedetti-Pichler and Schneider at Queens College and Stock at the University of Connecticut taught microscale techniques and published numerous papers and texts.⁵

These programs, however, were not widely adopted. This was due to several factors:

- The concerns we have for the environment today were not in vogue before 1970.
- There was little concern for the laboratory air quality, as risks associated with chemical exposure were not known, or seriously underestimated.
- Costs of chemicals were low and recycling was not considered as an option.
- Cleanup consisted of all waste being dumped down the drain.
- Only small numbers of students who could handle the manipulations were involved in the upper level courses where the microscale techniques were used.
- The lack of analytical balances that permitted rapid weighing of chemicals placed a monumental time restriction on the utility of microscale techniques.

Thus, microanalysis was highly delicate, tedious, time consuming and specialized. The technique was confined to a very limited and restricted area of research in graduate schools and industrial laboratories. In order for new techniques to become widespread, there must be practical reasons for their adoption, and it must be technologically possible to implement them. It was not until the 1980's, when environmental concerns had risen to the forefront and the electronic milligram balance became available so that using microscale experiments at the introductory level of instruction became a reality. A complete overview of the microscale laboratory development has been published.⁶

⁵ See (a) J.T. Stock, *J. Chem. Educ.*, 1990, 67, 898; (b) J.T. Stock, M.A. Fill, *J. Chem. Educ.*, 1953, 30, 296 and references therein; (c) S.W. Breuer, *Educ. in Chemistry*, 1991, May, 75.

⁶ See R.M. PIKE, Z. Szafran, M.M. Singh, "The Microscale Chemistry Laboratory" in: *Pollution Prevention and Waste Minimization in Laboratories*, P.A. Reinhardt, K. L. Leonard, P.C. Ashbrook (eds.), CRC Press, Inc., Boca Raton, FL, 1996, Chap., 13, p. 207.

What are the end results and the major advantages of this unique revolution? By reducing the amount of chemicals used in any type of chemical laboratory, several positive results occur:

- Very little or no toxic chemical waste is produced. Chemical pollution is therefore largely eliminated at the very source.
- The exposure to toxic materials is sharply reduced both for the chemist or technician directly working with the chemicals, and for individuals living downstream or downwind.
- Risks of fire and explosion are nearly eliminated.
- Air quality is markedly improved.
- On-site recycling becomes a more viable option, reducing waste production even further.
- Storage facility needs are reduced. Reduced chemical usage translates into less chemicals being purchased, less needing to be stored, and less decomposing due to short shelf-life.
- Operational costs are reduced in many ways. There is less expenditure for starting materials, for storing bulk chemicals, for waste disposal, for liability insurance and coverage, and workmen's compensation costs due to chemically-related illness.
- Expensive retrofit of existing laboratories to meet current air quality regulations is avoided.
- Once too costly experiments can now be done.
- Open-ended experimentation by the student is possible and teachers can make creative assignments over a wider range of chemical transformations.
- Governmental regulations and standards are readily achieved.
- The approach is ideally suited for qualitative as well as quantitative experimentation.
- Better trained students result. The main reason being that attention to detail is a must to be successful at this level of operation. The laboratory is now an exciting and fun place to be.

And what of the future? There is no turning back to the old ways of introductory laboratory instruction. The point has been proven beyond any reasonable doubt. The microscale technique has revolutionized the instructional laboratory.

There is an important point to be made, however. Students should be exposed to some macroscale level experimentation. Prior training at the micro level only enhances the ease and increases the ability of the student to make this transition.

The micro approach is also being adopted by the industrial sector in ever increasing ways. For example, the new thrust in industry is to implement the concept of "Green Chemistry" which focuses on the design, manufacture, and use of chemicals and chemical processes that have little or no pollution potential or environmental risk.⁷ The marriage of microscale techniques with this approach offers a double-barreled focus as a means to solve many of our environmental problems.

We continue to look forward with increasing enthusiasm and vision.

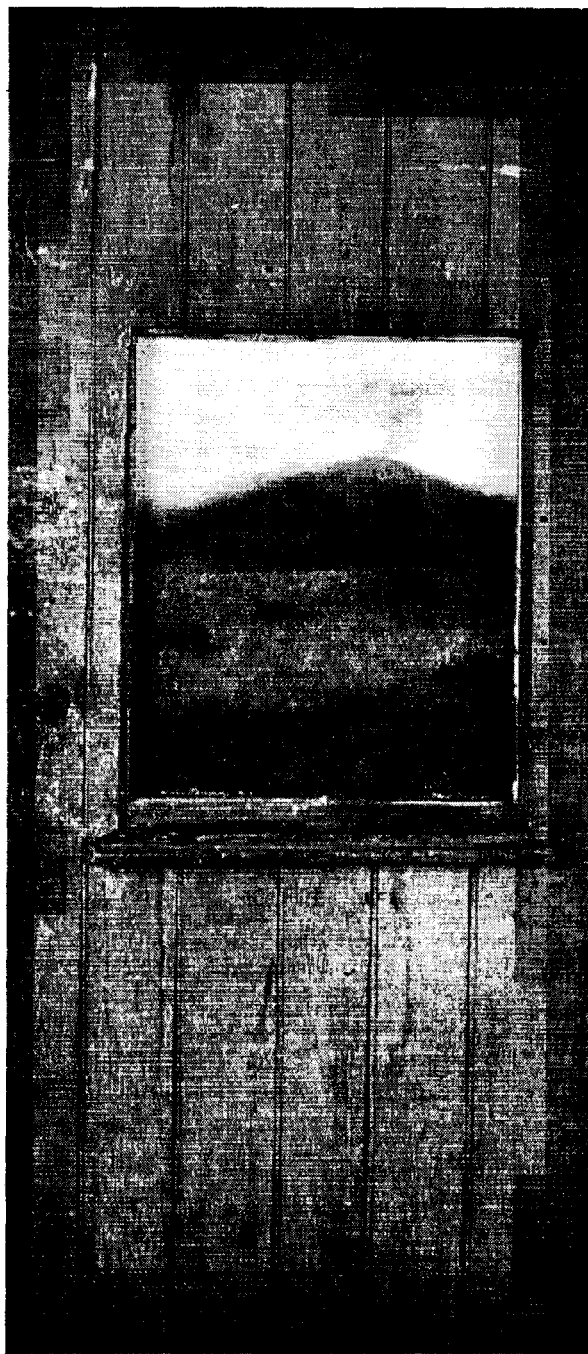
At a seminar presented at Merrimack College in 1990, Professor Terrance Morrill of the Chemistry Department of Rochester Institute of Technology, and co-author of a recent microscale organic laboratory text, stated "the introduction of the microscale concept into the introductory organic laboratory is the most significant advancement in chemical education in the last 50 years".⁸ As we have outlined here, the concept of working with micro-quantities of materials has spread to all types and classes of introductory science and chemical laboratories. Is the statement by Professor Morrill true? Time can only answer the question. We hope that you will become part of this great adventure and continue to move the concept forward. It all comes down to attitude. Do you view this concept, with its many benefits, in a positive light or do you say "we've never done it this way before", and keep on in the same old way?

We urge you to explore new areas in which to apply the approach, develop new techniques and above all, expose your students to the exciting world of microscale Chemistry. As stated by G.A. Crosby—"Microscale Chemistry is not merely a downsizing of macroscale experiments to smaller dimensions and amounts: it is proving to be the catalyst for new and exciting approaches to teaching Chemistry in the laboratory".⁹ This is where the future lies, if Chemistry as we know it today, is to reach to new heights for the good and betterment of us all.

⁷ See P.T. Anastas, T.C. Williamson (eds.), "Green Chemistry", ACS Symposium Series 626, American Chemical Society, Washington, DC, 20036, 1996.

⁸ T.C. Morrill, Private communication, 1990.

⁹ G.A. Crosby, *Chemunity News*, 1994, 4, 3.



Carol Anthony, *New Mexican Door*; Mera Hickey, 1991. Tomado de *The Sciences*, marzo-abril, 1999, p. 21.

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