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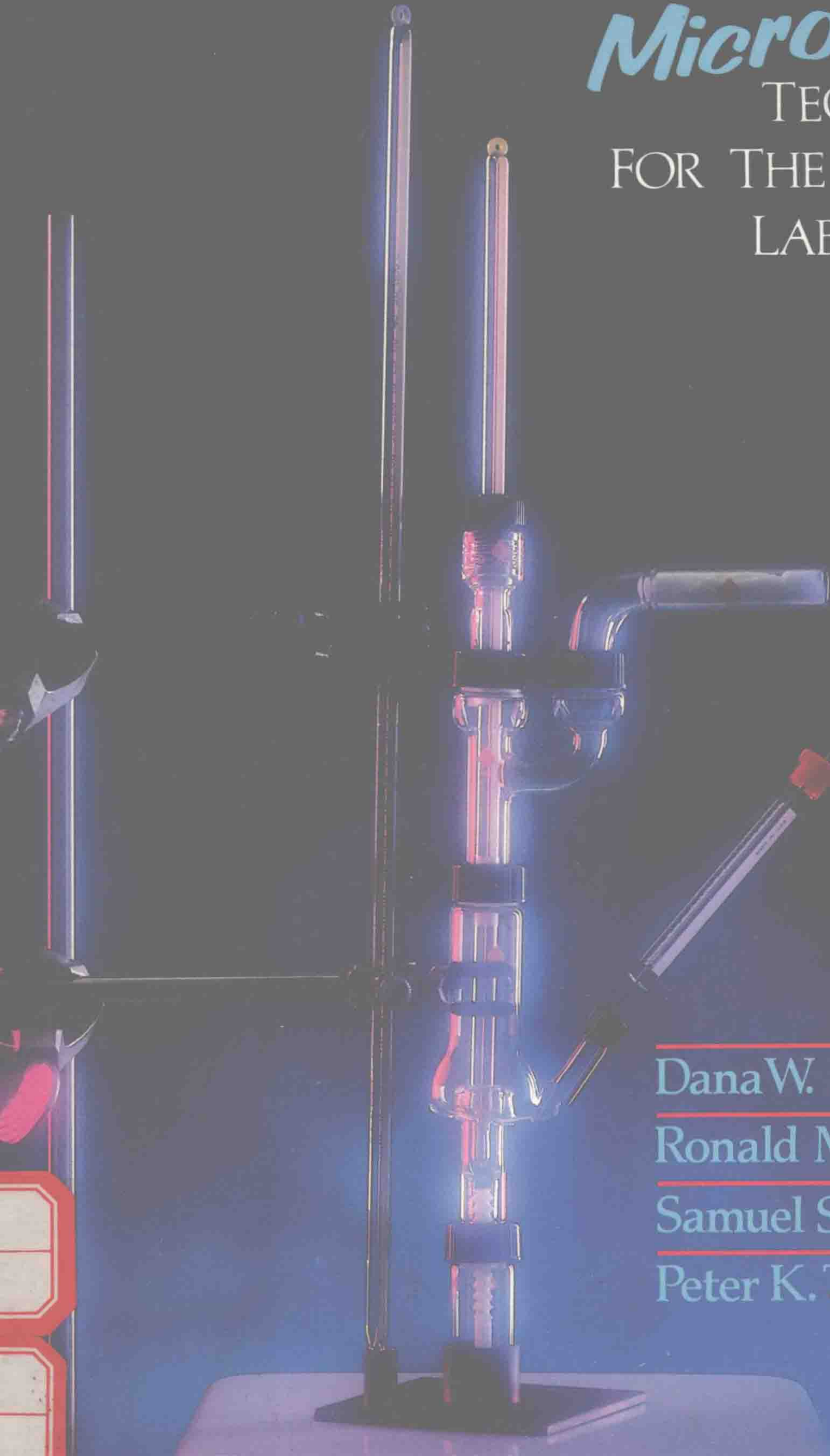

Dana W. Mayo
Ronald M. Pike
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Dana W. Mayo
Bowdoin College

Ronald M. Pike
Merrimack College

Samuel S. Butcher
Bowdoin College

Peter K. Trumper
Bowdoin College

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All experiments contained herein have been performed several times by students in college laboratories under the supervision of the authors. If performed with the materials and equipment specified in this text, in accordance with the methods developed in this text, the authors believe the experiments to be a safe, valuable educational experience. However, all duplication or performance of these experiments is conducted at one's own risk. The authors do not warrant or guarantee the safety of individuals performing these experiments. The authors hereby disclaim any liability for any loss or damage claimed to have resulted from or related in any way to the experiments, regardless of the form of action.

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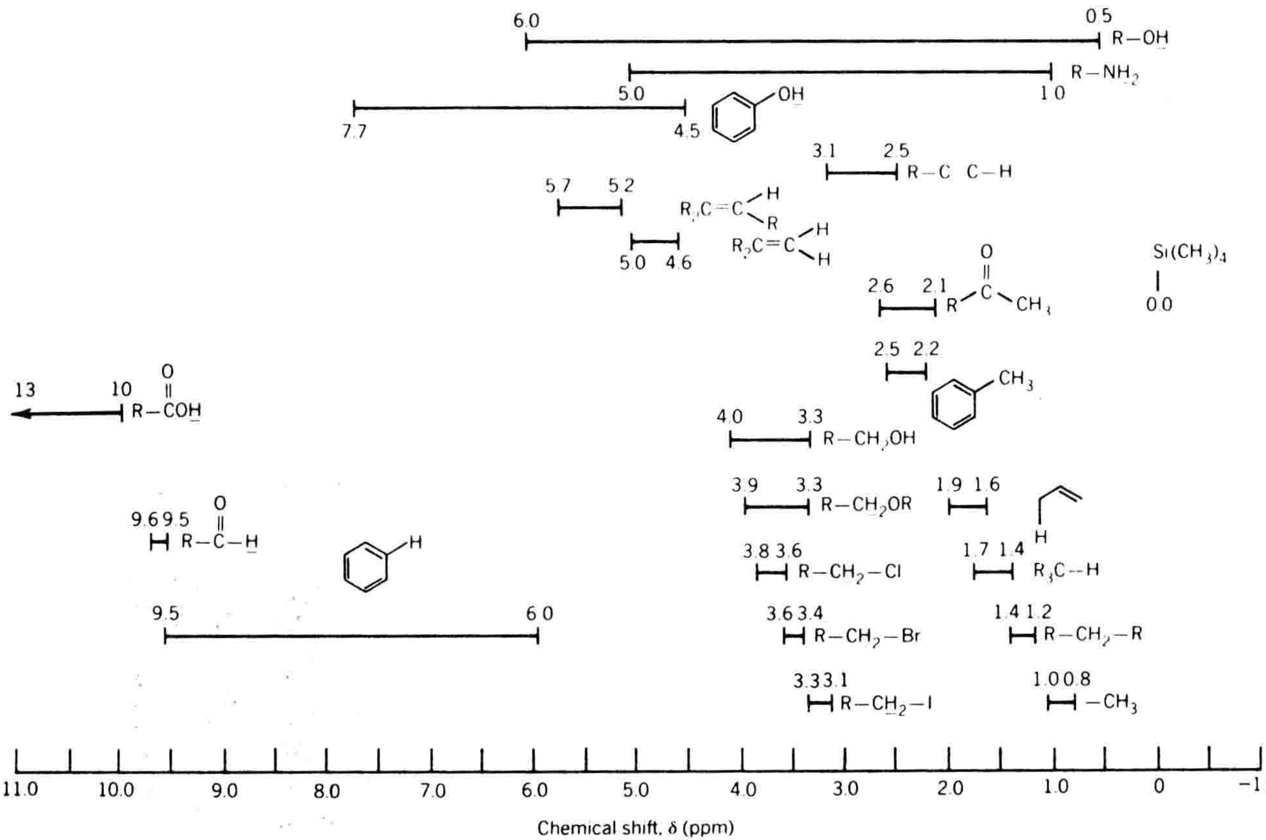

NMR H[1] *chemical shifts. From* Zubrick, J. W. *The Organic Lab Survival Manual,* 2nd ed.; Wiley: New York, 1988. (Reprinted by permission of John Wiley & Sons, New York.)

To Jeanne d'Arc, Marilyn,
Sally, Micòl, and Ceinwen.

About the Authors

DANA W. MAYO holds the Charles Weston Pickard Professor of Chemistry Chair at Bowdoin College. A former Fellow of the School for Advanced Study at Massachusetts Institute of Technology and a Special Fellow of the National Institutes of Health at the University of Maryland, he received his Ph.D. in Chemistry from Indiana University. Professor Mayo is Director of the Bowdoin College Summer Courses in Infrared Spectroscopy. His research interests include the application of vibrational spectroscopy to molecular structure determination, natural products chemistry, and environmental studies of oil pollution.

RONALD M. PIKE is Professor of Chemistry at Merrimack College. He received his Ph.D. from Massachusetts Institute of Technology. His main research interests involve the synthesis of organofunctional silanes and related silicone polymers. He is the author of numerous papers and patents in this area and is a coauthor (with Szafran and Singh) of *Microscale Inorganic Chemistry: A Comprehensive Laboratory Experience.* Professor Pike was previously associated with Union Carbide Corporation and the Lowell Technological Institute. He has been a Visiting Charles Weston Pickard Professor of Chemistry at Bowdoin College and a Visiting Professor at the U.S. Military Academy, West Point, NY.

SAMUEL S. BUTCHER is Professor of Chemistry at Bowdoin College. His research interests lie in the areas of atmospheric chemistry and air pollution. He is the coauthor of a textbook on air chemistry and has written several papers on air pollution and the effects of small wood-burning stoves. He has taught a wide range of undergraduate chemistry and science courses for nonspecialists. Professor Butcher received his Ph.D. in Chemistry from Harvard University.

PETER K. TRUMPER is Assistant Professor of Chemistry at Bowdoin College. He received his Ph.D. from the University of Minnesota.

His research interests include organic synthesis, stereochemistry, and the chemistry of sulfoxides.

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DANA W. MAYO and RONALD M. PIKE jointly received the James Flack Norris Award (1988) for outstanding achievement in the teaching of chemistry, and the John A. Timm Award (1987) for their work in developing the microscale instructional program. They both have received the Catalyst Award of the Chemical Manufacturers Association (DWM–1989, RMP–1990). Together with Samuel S. Butcher, they were corecipients of the first Charles A. Dana Foundation Award (1986) for Pioneering Achievement in Health and Higher Education and of the American Chemical Society Division of Chemical Health and Safety Award (1987). They also are the coauthors of *Microscale Organic Laboratory* (2nd Edition, 1989), the original microscale introductory organic laboratory text.

Preface

The widespread application of microchemical techniques to the introductory instructional laboratories began in the 1980s.[1] Since then the pace of microscale conversion has accelerated with the effects of this acceleration being experienced throughout chemical education and elsewhere. Microscale programs have been shown to be clearly superior in acquainting students with the operation of the chemical laboratory, but perhaps, more importantly, they also are having a significantly positive impact on laboratory morale. At a recent seminar it was stated that "the development of the microscale concept in the environs of the introductory organic laboratory is perhaps the most significant single advancement in chemical education in the last half century."[2] Beyond the pedagogic benefits of miniaturization, the attendant improvements to safety, air quality, and waste generation can only be described as spectacular.[3] This environmentally sound style of laboratory instruction unquestionably will become a preferred educational pathway in the teaching of chemistry as the twentieth century draws to a close and the twenty-first century begins.

The decade of the 1990s will be a period of active search for the right balance of experimental composition in instructional laboratories. One further point also has become very clear. Microscale experiments will have to play a major role in the introductory organic program, if the organic laboratory is to survive rapidly escalating operating costs and liability demands. The primary role we envision for this textbook is to provide flexibility to the instructor who wishes

[1]D. W. Mayo, R. M. Pike, and S. S. Butcher. *Microscale Organic Laboratory Preliminary 1*, John Wiley & Sons, Inc., 1985.

[2]Terence C. Morrill (RIT), Department of Chemistry, Merrimack College, April, 1989.

[3]D. W. Mayo, S. S. Butcher, R. M. Pike, C. M. Foote, J. R. Hotham, and D. S. Page, *J. Chem. Educ.* **1985,** *62,* 149.

to introduce a more personalized set of microscale experiments than is possible with the current microscale laboratory texts. Furthermore, *Microscale Organic Laboratory Techniques* should be adaptable for use in conjunction with any of the currently available macroscale laboratory program guides. This book gives the instructor the opportunity to mix an almost infinite variety of miniaturized instruction with as much conventional-scale work as he or she desires. The detailed descriptions of microscale techniques allow the instructor to weld them into a program in the most pedagogically profitable fashion.

The number of publications that describe microscale techniques and experimental reaction conditions has exploded over the past several years. Bowdoin College has published for the last four years a newsletter, *Smaller is Better,* and the *Journal of Chemical Education* has recently introduced a monthly experimental section. Both the newsletter and the experimental section are devoted entirely to this subject. From the worldwide interest in microscale techniques it is evident that many educational institutions will wish to draw from the current literature to formulate a set of microchemical experiments that fits their particular situation. With this premise in mind, this text was conceived; that is, its purpose is to provide a straightforward presentation of microscale techniques and procedures that both the beginning and the more advanced student will readily appreciate. *Microscale Organic Laboratory Techniques* is designed to function as a reference text that can serve the instructor, student, and research chemist in a variety of ways. The scope of discussion runs from detailed descriptions of the basic manipulative techniques to the more advanced methods used in the research laboratory. A number of carefully chosen examples that illustrate these techniques are included where it was thought that amplification would be helpful.

As students who have experienced the microscale approach begin to enter graduate and industrial laboratories in significant numbers, it is apparent that they are going to have a major impact on the manner in which chemical research and development are carried out in this country. Not only will their effect influence the scale at which experiments are conducted but it is going to significantly alter the philosophical approach by which concepts, projects, and products are launched in the industrial world.

Many of today's practicing chemists have little or no background at the microscale level. For premicroscale-trained chemists to make a smooth transition through this conversion period, they must have available a sound reference text that clearly illustrates the procedures and techniques employed in the microscale world. This was the second of the major objectives in composing this text. *Microscale Organic Laboratory Techniques,* outlines in detail many of the key manipulative procedures required for the successful operation of the miniaturized research laboratory. Thus, we hope that this text will function as a ready-technique reference handbook and that it will go a long way toward bridging the gap between the novice and the more experienced microscale chemist.

Microscale Organic Laboratory Techniques (MOLT) is primarily based on the procedures that evolved during the development of the microscale organic laboratory programs at Bowdoin and Merrimack and which are described in the original laboratory text, *Microscale Organic Laboratory* (2nd. Edition, Wiley © 1989). As discussed above, MOLT is designed to give a working view of the manipulative techniques and theory that one must master when undertaking experimental chemistry at the microscale level. The areas discussed are principally techniques that are used in the preparation, isolation, purification, and characterization of organic reaction products and naturally occurring materials. A number of the procedures and apparatus involve systems uniquely associated with the development of our particular program. Several examples are fractional distillation employing *bottom-driven* spinning-band distillation columns, preparative gas chromatography utilizing a unique collection device, recrystallization techniques that include a modified Teflon® Craig head, determination of liquid densities using "microcaps," ultra-micro boiling point and evacuated melting point determinations, glassware employing the gas-tight grease-free ground-glass O-ring cap seal. Separate chapters are devoted to a detailed introduction to NMR and IR spectroscopic techniques. The treatment of these areas includes theory, application, instrumentation, and sampling. In addition, there are discussions of ^{13}C, and two-dimensional NMR plus Fourier transform infrared spectroscopy.

We are indebted to the ever-expanding enthusiastic acceptance of a program that in its implementation requires a major commitment of time, effort, and financial support. Clearly, the results speak for themselves. Once operational, the microscale organic laboratory program is a major improvement in conveying the beauty and excitement of experimental organic chemistry to the introductory student. At a time when the very existence of these organic laboratory programs has been threatened, the microscale organic laboratory has made this aspect of the subject one of its most attractive facets.

Many people have aided, guided, and encouraged us over the past decade. We are particularly indebted to those earlier investigators who were deeply involved in establishing the educational side of miniaturized organic chemistry: F. Emich, and F. Pragel in Austria, and N. D. Cheronis, L. Craig, R. C. Fuson, E. H. Huntress, T. S. Ma, A. A. Morton, F. L. Schneider, R. L. Shriner, and J. T. Stock in the United States. We acknowledge the contributions to the development of microscale techniques that have come from all sides: contributors to the *Smaller is Better* newsletter, participants at the Bowdoin College Summer Institutes, and our students at Bowdoin and Merrimack. In particular, Robert Hinkle (Bowdoin '86), now at the University of Utah, made the seminal suggestion that led to the development of the enormously powerful bottom-driven spinning-band distillation systems. Larry Riley and J. Ryan of the ACE Glass Company and Robert Stevens of J. J. Stevens were responsible for the magic surrounding the Teflon® Craig head. Robert Matheau of GOW–MAC was quick to adapt a student–model gas chromatograph to accept the Bowdoin-developed GC collection system. We appreciate very much the encouragement and guidance of Dennis Sawicki, our chemistry editor at Wiley, and the thoughtful contributions of Dawn Stanley, our book designer, while transforming our manuscript into the final text. As in the past, our old friends Paulette Fickett, Lauren Bartlett, Judith Foster, Henry Horner, and Thomas Tarrent have continued to make major contributions to the evolution of the program. We acknowledge the continued support of the National Science Foundation to our Microscale Summer Institute and to the PEW Charitable Trusts for support of two summer institutes, in addition to their support of the construction of a new microscale organic laboratory at Bowdoin College. D. W. M., R. M. P., and S. S. B. are pleased to have Peter Trumper's high-field NMR expertise begin to weld itself into the educational side of microscale organic chemistry. Clearly many exciting advances will be occurring in the near future in this area.

The success or failure of the bench research chemist, to a very large extent, depends on an individual's ability to engage a vast array of laboratory skills while bringing about the solution to challenging experimental problems. It is our hope that this book will be a friendly roadmap for those who, in the future, will be exploring and expanding the exciting world of organic chemistry while pursuing microscale techniques.

December 1990
Brunswick, Maine

DANA W. MAYO
RONALD M. PIKE
SAMUEL S. BUTCHER
PETER K. TRUMPER

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