INTEGRATED EXPERIMENTAL CHEMISTRY

Principles and Techniques

Volume 1

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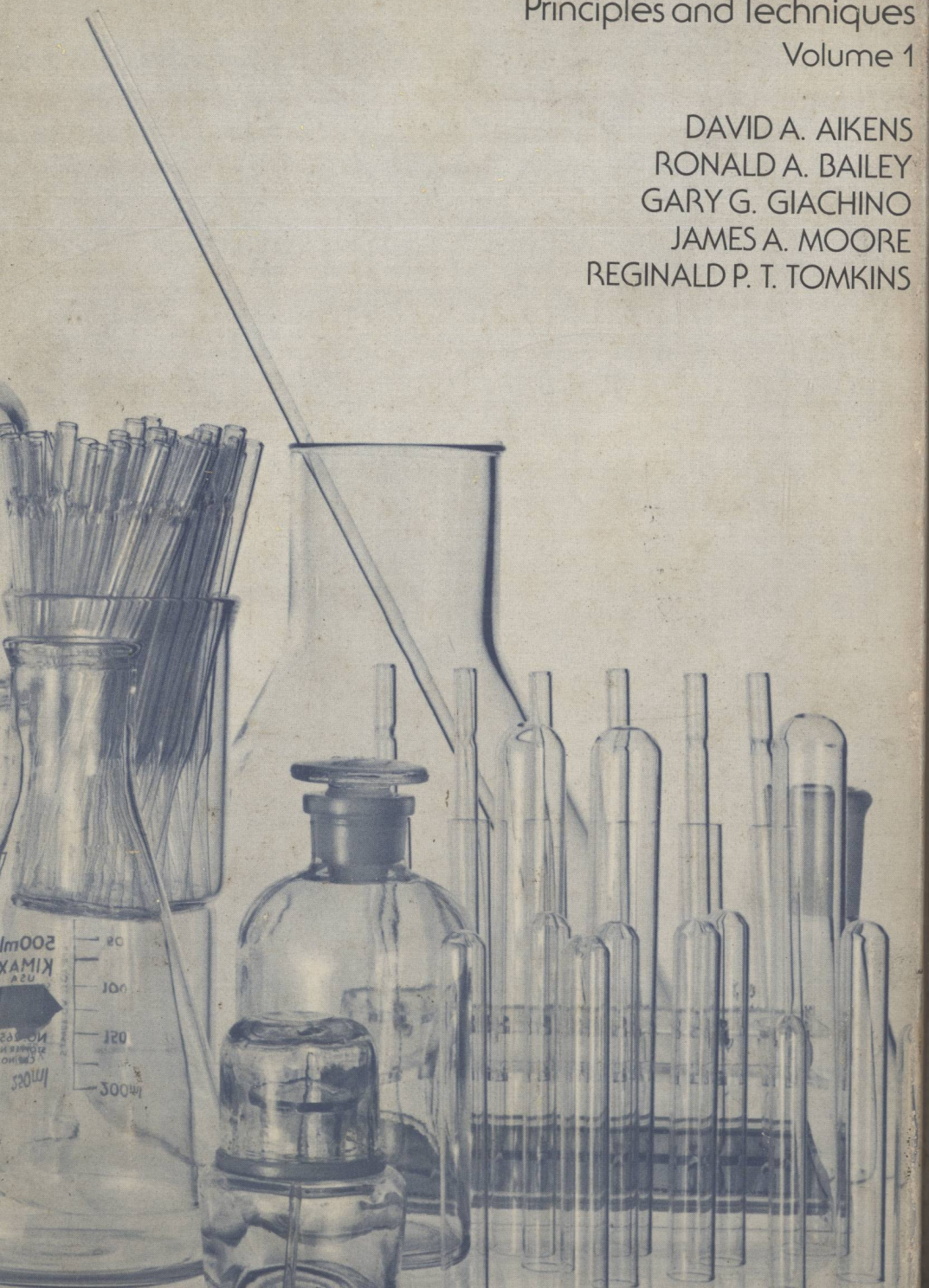

Integrated Experimental Chemistry

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Preface

A number of chemistry departments have concluded that the undergraduate chemistry laboratory program can be improved by replacing the traditional compartmentalized laboratories in analytical, organic, inorganic, and physical chemistry with an integrated laboratory program. In comparatively few of these approaches, however, has integration been complete, and usually one or more of the traditional areas has not been included. Other approaches have been based on specialized or costly experiments. The lack of a comprehensive textbook for a unified laboratory has certainly been a barrier towards progress in the adoption of this concept.

It has become apparent that the modern practice of chemistry demands that students become familiar with a variety of instrumentation. The use of techniques such as infrared, visible-ultraviolet and nuclear magnetic resonance spectroscopy, gas chromatography, and liquid chromatography are becoming routine in industrial, academic, and governmental laboratories.

In 1971, the Chemistry Department at Rensselaer Polytechnic Institute initiated a four semester unified laboratory program which combined all of the required chemistry laboratory courses taken by chemistry majors after the freshman year (analytical, instrumental, organic, and physical chemistry). The sequence is based on two 4-hr laboratory periods a week in each semester, and a discussion period each week in the first two semesters. This two-volume text is the result of the laboratory manuals developed for this program. For the most part the experiments are based on low-cost chemicals and equipment, but they are strongly oriented towards early and routine use of instruments in parallel with the techniques of wet chemistry. All instruments employed with these experiments are intended for "hands-on" use by the student with only normal supervision. We have not included discussion of instrumental techniques where such use has been impractical – for example, mass spectroscopy. The program was developed around equipment already used in the "classical" laboratory program; and contrary to general opinion we found that adoption of the integrated laboratory format did not cost significantly more than individual laboratories of the same level.

The program is based on individual experiments of 3 to 20 hr or more in length, which may be arranged in a variety of ways to provide flexibility.

Experiments that are logically related or that form a sequence can be chosen to give a theme for a semester's work. The choice of appropriate experiments will depend upon the previous and simultaneous theory courses. By focusing on experimental chemistry as a whole rather than on isolated segments of laboratory work, by providing experiments that require the students to exercise judgment and to make decisions with regard to exact procedures, by including optional experiments and projects, and by having realistic laboratory situations and flexible scheduling, this program is advantageous in developing maturity and independence as well as technical laboratory skills.

Volume 1 of the text describes the experimental techniques likely to be encountered by the undergraduate chemistry student, gives some mention of more advanced techniques that will be helpful in project work, and provides references to more specialized discussions. The theoretical principles of each technique are described only briefly as needed, and for a detailed treatment of theory other sources must be utilized, such as the text-books used in the corresponding theory courses. It seems unnecessary and unwieldy to duplicate such material in a book of this nature. Volume 1 also discusses keeping records and writing reports, the treatment of errors, data sources, and laboratory safety. Volume 2 contains a selection of experiments appropriate to an integrated laboratory program. Volume 1 should be useful to those who have their own selection of experiments as well as to those who elect to use the experiments of Volume 2.

The development of this text has been aided by the input of many students at Rensselaer, and by contributions from our colleagues, particularly N. F. Hepfinger, H. F. Herbrandson, R. L. Strong, and J. W. Zubrick. We wish to acknowledge the support from G. J. Janz and K. T. Potts who, as Department Chairmen, encouraged the implementation of this program.

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Introductory Remarks to Students

The importance of a good laboratory program lies in the fact that chemistry is fundamentally an experimental science; theories are necessary to correlate experimental observations and to guide the development of new experiments, but they are valid only in so far as they can do this. Most students majoring in chemistry will spend a significant portion of their future careers involved with laboratory work; either as direct participants, or as those who must use and evaluate experimental results. Consequently, a good understanding of how these results are obtained, and a sensitivity for their probable validity and reliability, is important. Those not continuing in chemistry but branching into another field of science will find that much of the approach described in this book will still be applicable. The program covers a broad area of chemistry, and it presents the field as a whole rather than as isolated segments, thus providing a flexible background for a variety of future interests.

The basic concept of an integrated or unified laboratory program is the incorporation in a logical fashion of several techniques and procedures into one experiment. For example, an experiment may involve a particular synthetic procedure, use various methods of purification and characterization, include some physical property measurements on the product and, where appropriate, study the thermodynamics or kinetics of the reaction. This approach emphasizes that techniques should not be regarded as being specific to the various traditional areas of chemistry, i.e., organic, physical, analytical, or inorganic chemistry, but rather that they can be applied to study problems in any area of chemistry, under appropriate circumstances.

The experiments described in this text stress the relationships among various techniques and measurements, and they present both classical and instrumental techniques in realistic applications. An early introduction to instrumental techniques such as infrared spectroscopy, gas chromatography, ultraviolet and visible spectroscopy, and nuclear magnetic resonance spectroscopy, is a key feature of these experiments. More sophisticated experiments demonstrating advanced uses of these techniques are then introduced once this foundation has been laid.

The main goals of the program are to provide:

manipulative skill with a wide variety of procedures and equipment;
wide experience with properties and characterization of chemical systems;
early and routine use of instruments that are common in research and industrial chemistry laboratories;
experience with the application of a wide variety of instruments and techniques to the solution of a particular problem;
practice in the selection of conditions or techniques for solving a problem;
reinforcement of key theoretical principles through repeated encounters at progressively more advanced levels;
awareness of limitations and errors in experimental procedures;
development of critical judgment;
development of creativity by encouraging individual approaches to the solution or problems.

In so far as possible, a given experiment will combine several of these objectives. However, before properties or principles can be examined in a serious and sound manner, a certain competence in experimental techniques must be acquired, and the initial experiments stress the acquisition of basic laboratory skills.

The total text is organized into two volumes. Volume 1 describes general laboratory techniques and procedures with emphasis on the technique rather than on the theoretical principles. Only enough theory is given to provide a basic understanding of the technique. It is assumed that students will have taken theory courses appropriate to the assigned experiments, and are familiar with text books which deal more thoroughly with theoretical principles. The use of several information sources is an important educational goal. Volume 2 contains a collection of experiments suitable for a unified laboratory.

Several unknown compounds or mixtures must be identified, either as parts of individual experiments or as separate projects. A chapter on the identification of unknown compounds is included in Volume 1, and some specific functional group tests are described in individual experiments. However, a supplemental text on qualitative organic analysis is recommended to provide additional test procedures. In a few experiments, specific tests are called for that are not described in this text, and that require the use of a supplemental text or the resources of the library.

The experimenter must be aware of the safety precautions necessary in the laboratory, and must read the chapter on safety before beginning laboratory work. All safety regulations must be understood and obeyed. Organized work in which the procedures have been thought out carefully is the best safety precaution.

Contents of Volume 1

Contents of Volume 2