ENVIRONMENTAL LABORATORY EXERCISES
FOR INSTRUMENTAL ANALYSIS AND
ENVIRONMENTAL CHEMISTRY
ENVIRONMENTAL LABORATORY EXERCISES FOR INSTRUMENTAL ANALYSIS AND ENVIRONMENTAL CHEMISTRY

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To my parents for nurturing
To my advisors for mentoring
To my students for questioning
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My most vivid memory of my first professional job is the sheer horror and ineptitude that I felt when I was asked to analyze a hazardous waste sample for an analyte that had no standard protocol. Such was life in the early days of environmental monitoring, when chemists trained in the isolated walls of a laboratory were thrown into the real world of sediment, soil, and industrial waste samples. Today, chemists tend to be somewhat better prepared, but many still lack experience in developing procedures for problematic samples. My answer to this need for applied training is a book of laboratory experiments aimed at teaching upper-level undergraduate and graduate chemistry students how to analyze “dirty” samples. These experiments can be taught under the auspices of a standard instrumental analysis course or under more progressive courses, such as environmental chemistry or advanced analytical environmental techniques.

In preparing this book, I have kept in mind a number of chemical and analytical considerations, some stemming from fundamental principles taught in every chemistry department, others specific to environmental chemistry. First, chemists planning to work in the environmental field need to be aware of the uncompromising need for explicit laboratory documentation. Chemistry departments start this life-long learning exercise in general chemistry, where we tell students that any classmate should be able to pick up his or her laboratory notebook and repeat the work. Environmental chemistry takes this training one step further in that the experiments and their documentation must also be completed in a manner that is legally defensible. By legally defensible, I mean ready to serve as courtroom evidence, as almost any laboratory monitoring, no matter how routine, can easily become evidence to prosecute an illegal polluter. Thus, laboratory notebooks must be maintained in a standardized format (subject to state or federal authorities and discipline); if they are not, cases may be
dismissed. The introduction to this manual contains a list of commonly accepted documentation procedures. They are arranged so that instructors can select which level of documentation is suitable for their course.

A second feature of this manual is that it is designed to be a complete, stand-alone summary of a student’s laboratory work. In the student version of the laboratory manual, each procedure contains background information, safety precautions, a list of chemicals and solutions needed, some data collection sheets, and a set of blank pages for the student to compile results and write a summary of findings. Thus, when each experiment is finished, students have a complete summary of their work that can be used as a laboratory portfolio during interviews at graduate schools or with potential employers.

A third theme, presented early in this book, is statistical analysis. Although many students entering environmental chemistry or instrumental analysis have briefly studied linear regression and Student’s t test, a more rigorous treatment of these topics is needed in laboratories dealing with instrumentation. As I tell my students, few if any instrumental techniques yield absolute numbers; all instruments have to be calibrated to some extent, and the most common approach is a linear least squares regression. One of the first exercises that I conduct in my classes is to have students build a spreadsheet to perform linear least squares analysis and Student’s t test. I have found that students understand data analysis techniques significantly better after this spreadsheet exercise, as opposed simply to quoting numbers from the regression of a calculator. An electronic copy of these spreadsheets (which I have students replicate) is included with the instructor’s edition, and the spreadsheets can be used throughout the semester for a variety of instruments.

Fourth, the laboratory exercises in this manual are designed to teach environmental chemistry and instrumental analysis simultaneously. The experiments are organized by sample media into sections of air, water, hazardous waste, sediment/soil, and wet techniques, and the manual includes a set of pollutant fate and transport simulation exercises, which are becoming more and more necessary in environmental chemistry courses. The laboratory experiments emphasize sampling, extraction, and instrumental analysis. Interactive software packages for pollutant fate and transport simulations, Fate and the pC-pH simulator, are included with the text.

Compiling the experiments for this manual has been a very educational experience for me, as I have reflected on which experiments work best in which setting. This information is given in the notes to the instructor. All of the experiments have been used in my courses, either environmental chemistry or instrumental analysis. More important for instructors using this manual, most experiments have a sample data set of the results expected, which is posted on the Wiley website. Each year I find these sample results most helpful in troubleshooting laboratories and identifying student mistakes.

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F.M.D.
This laboratory manual is designed for use courses in Instrumental Methods of Analysis and Environmental Chemistry. In fact, students from both of these courses were involved in the testing of these procedures. The procedures emphasize solution preparation, experimental setup, use of instrumentation, and evaluation of results. Given that not everyone is an environmental chemist, I have put together a list of experiments I use in instrumental analysis that are also used in environmental experiment. If you are unfamiliar with environmental chemistry I have included extensive background information on the environmental topic being studied and most chapters have a complete set of student data for your review (included in the on-line instructor’s information). Indeed, one advantage of using this manual is that I have found students to be very interested in learning from an environmental viewpoint.

For instrumental analysis, of course, I use the experiments that emphasize the instruments a bit more than the solution preparation. There are certain exceptions to this statement, for example Chapter 14 (The Determination of a Surrogate Toxic Metal in a Simulated Hazardous Waste Sample), which stresses matrix effects and technique specificity (chelation, activity, or concentration). The following is the general plan I used for the course on Instrumental Methods of Analysis. It is based on two 3-hour laboratory periods each week.

Chapters 1 and 2 are given as introductory material but I usually have students build a spreadsheet for the statistics chapter.

- UV-Vis spectroscopy: Chapter 10
- Infrared spectroscopy: Chapter 5
- Electrodes: Chapter 9 or 14
Atomic absorption or emission spectroscopy  Chapters 14 or 7
Gas chromatography  Chapters 6, 8, 11, or 16
High performance liquid chromatography  Chapter 13
Ion chromatography  Chapter 7
Mass spectrometry  any of the chromatography chapters

For environmental chemistry there are a variety of approaches. First, if you do not use this manual in a course in Instrumental Methods of Analysis you can select from all of the experiments. Second, if you use the approach given above for instrumental methods of analysis, there are still plenty of experiments left for use in environmental chemistry. I select from the following experiments.

Sampling  Chapter 2 (covered in lecture)
Mass balance, weighing and pipeting skills  Chapter 20
DO and BOD  Chapters 18 and 19
Global warming  Chapter 5
Environmental monitoring  Chapters 6, 8, 9, 13, 16, 21, or 22
Hazardous waste treatment  Chapter 12
Transformation reactions  Chapter 15
Distribution coefficients  Chapter 17
Chemical speciation  Chapter 23 (covered in lecture)
Pollutant fate and transport  Chapters 24 to 28 (covered in lecture)

An alternative is to design your environmental course completely around wet techniques.

Whichever way you choose to use this manual I hope that you will be satisfied with our efforts. We have done our best to provide student-tested procedures from an environmental perspective, detailed procedures for making solutions and unknown samples, example student data for troubleshooting and to supplement your students’ experimental data, two user-friendly software packages (The pH Simulator® and Fate®). Additionally, after you adopt the manual for use by your students you will have access to Wiley’s on-line resources for this manual and you will be sent The GC Tutorial and The HPLC Tutorial. The downloadable instructor’s manual can be obtained at http://www.wiley.com/wileyceda/wileytitle/productcd-0471488569.html. The latter two software packages are particularly helpful if students view them prior to attempting the chromatography experiments.