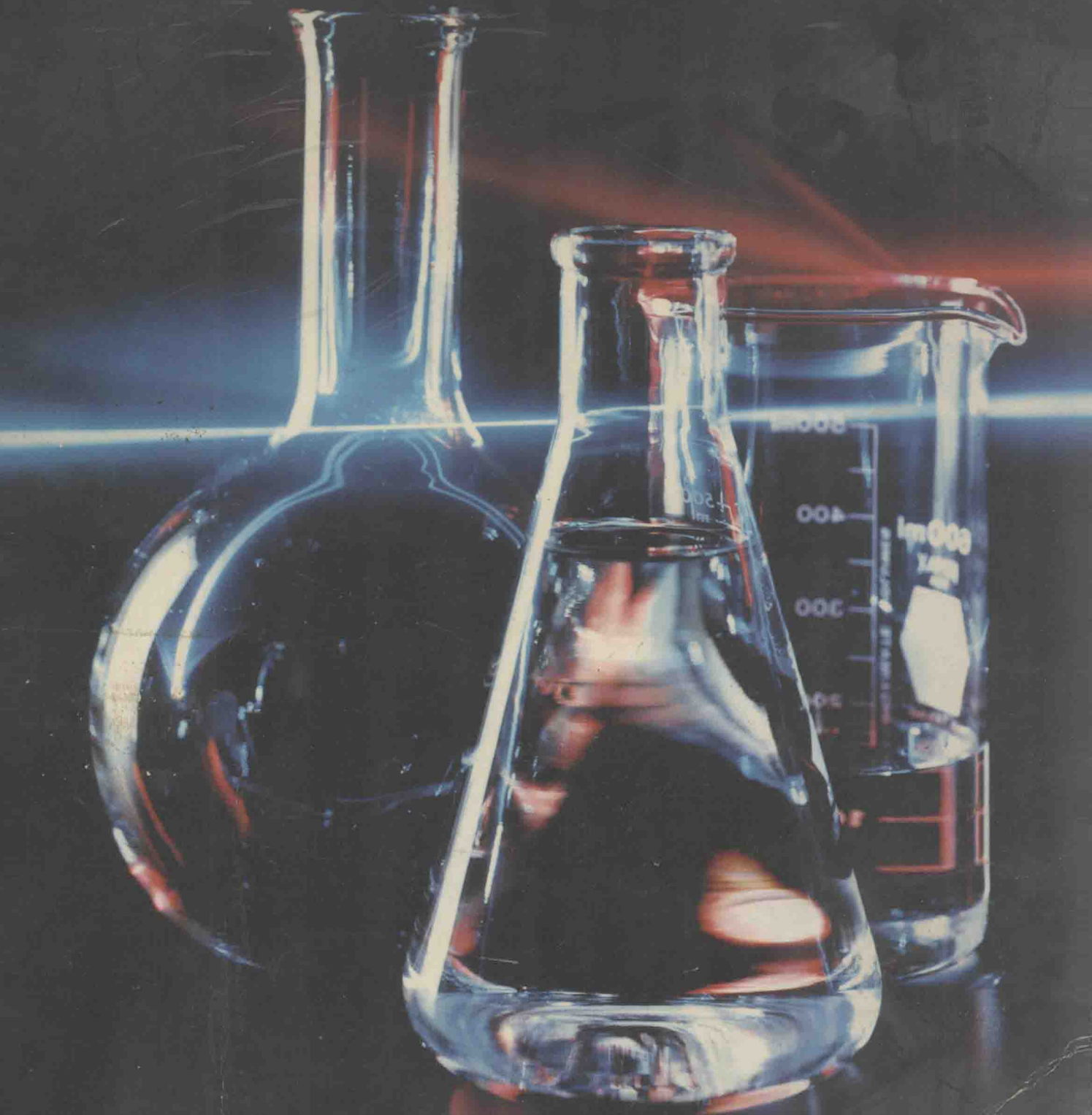
Qualitative Analysis
And Ionic Equilibrium

Schenk/Ebbing



Qualitative Analysis and Ionic Equilibrium

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Preface

This laboratory manual presents the traditional qualitative analysis scheme using explanations that are as modern as possible. It was written to be consistent with the notations and approach of *General Chemistry* by Darrell Ebbing (Boston: Houghton Mifflin, 1984). It can also stand alone or be used with most other texts. In contrast to some qualitative analysis texts, it does not deal with the chemistry of the *metals* or *nonmetals*; it stresses the chemistry of the *metal ions and anions*, both in general and as applied to qualitative analysis. For example, when discussing the chemistry of lead, we do not deal with the various ores of lead; instead we prefer to describe the various reactions of the lead(II) ion, including its ability to cause brain damage in young children (Section 5.3).

Standard Organization

The sequence of the chapters follows a fairly standard organization. Chapters 1 through 4 present introductory material that should be read and covered to some degree in lecture before, and during, the laboratory analyses. Each chapter is divided into *numbered sections* for easy reference and to enable the instructor to assign certain sections for reading only and certain sections for lecture background. Thus Sections 1.1, 1.2, and 1.4 in Chapter 1 might be assigned to the student for reading only. However, the instructor may wish to cover some of the colors of the ions in Section 1.3 in lecture, or to defer that section until the analysis of Group 2.

Chapter 2 is an *overview* of ionic equilibria. It can stand alone as a review of equilibria in a previous course, or it can be used to supplement any text's discussion. Most sections can stand alone, although a knowledge of buffers is important for Example 2.6 in the discussion of solubility equilibria (Section 2.4). In describing ionic equilibria, we have tried to avoid highly complicated situations, but we have attempted to be as honest as possible. For example, we have presented *stepwise* formation constants (Section 2.3) for the $Co(SCN)_4^{2-}$

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ion, but we have avoided combining these constants to calculate concentrations of each cobalt(II)-thiocyanate species. Finally, we have introduced the ionization of H_2S in Section 2.2, but have deferred calculations of $[S^{2-}]$ in H_2S solutions until Chapter 3.

Chapter 3 lays the foundation for the analysis of all five cation groups. It introduces the student to the concept of analysis for *groups of cations*, and then discusses in one section (3.1) all aspects of the ionization of H_2S including pH effects and precipitation of metal sulfides by pH control. The separation of Groups 2 and 3 is used to illustrate the pH control of precipitation. In Section 3.2, we define and discuss all of the *group reagents*, correlate all of the cations in our analysis on a periodic table (Figure 3.1), and summarize the separation of all 5 groups in a group separation scheme or flow chart (Figure 3.2). If desired, Sections 3.3 and 3.4 can be assigned for student reading only. In Section 3.3 we try to explain to the student what each unknown may consist of and indicate the possible combinations for the Group 1 unknown. In Section 3.4, we emphasize planning ahead for the laboratory and notebook. At the end of the chapter (page 55) is a sample notebook page set up for recording observations of the analysis of the first unknown (Group 1). Later in Chapter 5, Figure 5.4 (page 80) gives the student an example of a *partially completed* notebook page for that same first unknown.

Chapter 4 is a summary of lab techniques and approaches. It is positioned just ahead of the first cation group analysis (Group 1 analysis) in Chapter 5. It may be assigned for student reading, but we recommend that some of the material be used in one or more lab lectures. Although much of Chapter 4 should be covered before the first unknown, heating solutions and the water bath (Figure 4.4) are best reviewed right before the analysis of Group 2 in Chapter 6.

Chapters 5 through 11 describe the analysis of the five cation groups, the analysis of the anions, and the analysis of a general unknown, dry solid salt mixture, and/or an alloy. The periodic, electronic, and solubility features of the cations in each group are first discussed, followed by sections on individual cations or pairs of cations. Cations that have certain similarities are discussed together. For example, both copper(II) and cadmium(II) are discussed together in Chapter 6, iron(III) and cobalt(II) are discussed together in Chapter 7, and potassium and ammonium ions are discussed together in Chapter 9. These chapters end with a section on the group flow chart and analysis chemistry, a section on the analysis procedures, and finally a group report form.

The appendixes include answers to most even-numbered review questions and problems, tables of selected equilibrium constants, and directions for preparing reagents and unknowns used here at Wayne State University.

Developing Student Understanding of Analysis

First of all, we have tried to correlate the chemistry and color of the various ions to the periodic table (see Figures 3.1, 5.1, 5.2, 6.1, and 7.1). Second, we

have stressed the use of color in preliminary observations, precipitation, confirmatory tests, and flame tests. Color is first discussed in Section 1.3, and then reintroduced in the first section of every analysis chapter. Third, we have used ionic equilibria wherever possible to explain the chemical reactions. Finally, we have introduced a novel form of flow chart or "flow scheme" (for instance, see Figures 3.2 and 5.3). Each group or ion is always boxed off from the rest, and solutions are always shaded so that the eye can visually follow the separations easily and distinguish solutions from precipitates. When a group or ion is separated, it is written off to the right, leaving the rest of the groups or ions together at the left in the next row of boxes down. Each solution and precipitate is identified by numbers: S_1, S_2, P_1, P_2, and so on. The same order is maintained in each row of boxes. For example, in Figure 3.2, Group 5 is in the left box all the way down the figure.

Problem-Solving

In this laboratory manual, as in *General Chemistry*, the student is involved through examples, review questions, and problems. In the examples involving equilibrium calculations, the "concentration table" approach is used (see Example 2.2, for instance). An equilibrium equation is written and concentrations are tabulated below it. Review questions not only review the concepts in the chapter but also include the interpretation of typical analysis problems in Chapters 5 through 11.

The problem section at the end of every chapter is divided into groups of similar problems, each with its own heading. The problems within each group have been selected to include varying types and degrees of difficulty. Many problems have multiple parts, allowing students to gain confidence by solving several variations of a similar problem. Where possible, we have tried to include a number of problems involving equilibrium calculations at the end of each analysis chapter. This, we feel, has been a deficiency in a number of qualitative analysis texts.

Special Enrichment Topics

A few portions of the book have been separated into special enrichment sections. These may be assigned at the instructor's option. However, the student's understanding of the rest of the text will not be diminished if they are not read. These are Section 1.5 (Crystal Field Theory: A Review), Section 1.6 (Crystal Field Theory and Colors of Cations), Section 2.6 (Theoretical Calculation of the Equilibrium Constant), and Section 9.4 (The Cobalt Blue Optical Filter for Observation of Color). These sections have been used for many years in large classes at Wayne State University and are included with the hope that they may be useful to other instructors.

Acknowledgments

We would like to acknowledge the many fine people who have helped us in one way or another during the writing of this book. First, we must acknowledge Richard Hahn, who taught qualitative analysis for so many years here at Wayne State, and whose text *Inorganic Qualitative Analysis* influenced us both greatly. Many of our best approaches are good because of him. Perhaps our other approaches would have been better if we had been able to share them with him.

Then we wish to thank our fine reviewers, Muriel Bishop of Clemson University and H. Dale Warren of Western Michigan University. Joseph Oravec of Wayne State University checked the safety aspects as well as many other experimental details. The number of errors is infinitely smaller because of their fine efforts.

Most important of all, we both acknowledge the love and support of our families during this time. One of us [G.S.] especially wishes to dedicate this book to Lara Elizabeth Schenk (the most beautiful grandaughter born on November 18, 1983).

We are grateful to Brenda Schufelt and Marilyn Martin for initial testing of group procedures. And last we wish to thank all our friends here at Wayne State and elsewhere in the Detroit metropolitan area for encouragement and kind words.

G.H.S.
D.D.E.

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