THE ELEMENTS OF QUALITATIVE CHEMICAL ANALYSIS: WITH SPECIAL CONSIDERATION OF THE APPLICATION OF THE LAWS OF EQUILIBRIUM AND OF THE MODERN THEORIES OF SOLUTION. PARTS III AND IV LABORATORY MANUAL

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SPECIAL CONSIDERATION OF THE APPLICATION OF THE LAWS OF EQUILIBRIUM AND OF THE MODERN THEORIES OF SOLUTION

BY

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PARTS III AND IV
LABORATORY MANUAL

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PREFACE

THIS Laboratory Manual forms Parts III and IV of the author's "Qualitative Chemical Analysis." It is intended to be used in conjunction with Parts I and II, which are published as a separate volume and which contain the theoretical treatment of the subject. It will be sufficient to state here that Parts I and II include a discussion of the development and experimental foundations of the modern theories of solution; of the application of the laws of chemical and physical equilibrium to reactions used in analytical chemistry; of the theory of complex ions; of the electric theory of oxidation and reduction; and of the remaining important typical chemical actions used in analysis; all with special attention to substantiating the theoretical treatment by lecture experiments.

All of these topics are frequently referred to and intimately involved in the work of the laboratory course in qualitative analysis herewith presented.

The Laboratory Manual proper includes, as Part III of the author's book, a laboratory study of the analytical reactions of the common metal and acid ions and of the analysis of the various groups of these ions, as an introduction to the study and practice of systematic analysis. The laboratory work of this part (III) should be accompanied by classroom work on Part II of the first volume. The outline of systematic qualitative chemical analysis adopted forms Part IV of the book, and comprises the second half of the laboratory work provided for in this manual.

The author's viewpoint and the method of work recommended

are fully discussed in the preface to the first volume of the work. Teachers interested are referred to that preface.

In the same place acknowledgment is rendered to authors whose works have been consulted, and to friends and colleagues, whose assistance has greatly helped the author.

JULIUS STIEGLITZ.

CHICAGO, *September*, 1911.

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GENERAL LABORATORY INSTRUCTIONS

Apparatus. — 1. All apparatus must be cleaned and, when not in actual use, must be kept *clean* in the desk, ready for immediate employment. Distilled water is used for rinsing.

2. A *wash-bottle* and a set of *glass rods* (5, 7, and 9 inches long), rounded off at both ends, must be prepared.

Use of Reagents. — 1. Unless instructions call especially for the use of a *solid* reagent, the *solutions* of reagents on the desk-shelf and side-shelf are referred to in the instructions (but hydrogen sulphide is used as a gas, unless a solution of it is called for).

2. When instructions call for the use of the common acids and bases, without any specific qualification, the *dilute* solutions are intended to be used. *Concentrated* acids, etc., are used *only* when *specifically* called for.

3. The *concentrations* of all the reagents and solutions used are recorded in the table on pp. 143–147. In studying reactions, students should consult this table, so as to secure a basis for noting the intensity of reactions (precipitations, color reactions, etc.).

4. Reagents *must* be *filtered*, if they are not clear.

5. *Reactions* are tried, in test-tubes, with 5 to 6 c.c. of the solutions called for. A *small* excess of the reagent is used — that is, the reagent is added, until its obvious action (precipitation, solution, etc.) ceases. The common reagents represent rather strong solutions, and from 1 to 10 c.c. (usually 1 to 5 c.c.) of the reagent should be sufficient. An undue excess of reagents overloads solutions with matter foreign to the substance under examination and dilutes the latter.

Operations. — 1. Reactions are invariably carried out *at room temperature*, unless instructions specifically call for the application of heat. In the latter case the material under examination is *cooled for all operations subsequent to the one requiring heat*, unless specific instructions to the contrary are given.

2. *Filters* are used in funnels that are sufficiently large to leave at least a quarter of an inch of glass above the paper. A margin of more than one-half inch is avoided. The *size* of a *filter* is selected according to the *bulk* of the precipitate to be collected on it. *Traces* are collected on filters of 2 cm. diameter. The *centrifugal machine* is used with advantage in separating small precipitates from the great bulk of liquid, and the *filter-pump* is used for filtration.

3. All precipitates, which are collected on a filter, are *washed* twice, on the filter, with distilled water or with such reagent as the instructions call for.

Precautions. — 1. Potassium *cyanide* is a deadly poison. When it is used, special care must be taken to avoid breaking a glass vessel containing it, as

the absorption of the poison by a cut would be extremely dangerous. When a cyanide solution is acidified, the operation must be carried out under a hood, to prevent the inhalation of hydrocyanic acid fumes.

2. **Concentrated sulphuric acid** and concentrated nitric acid must be handled with great care. Concentrated sulphuric acid is *added to* water, alcohol, solutions, etc., very slowly and with constant cooling of the vessel used.

3. **Solid oxidizing agents** — nitrates, chlorates, permanganates, dichromates — must be used in small quantities, and care must be taken not to heat, or rub in a mortar, any mixture of such an agent with a strong reducing agent (organic substances, cyanides, ferrocyanides, ferricyanides, sulphides). When the analytical work demands the destruction of such a reducing agent (*e. g.* organic matter) by a powerful oxidizing agent, the laboratory instructions contain specific directions as to the process to be used.

Records. — 1. In the *study* of *reactions*, all *observations* should be *recorded* at once, briefly but accurately, on the blank leaves provided for that purpose. Since the laboratory instructions are largely put in the form of questions, the record of observations is necessary to make the text complete and to render it available in connection with the later work on systematic analysis. Answers to questions of theory, equations, etc., should be given in a separate notebook. The formulæ for common substances will be found in the lists of reagents, pp. 143–149.

2. The form of *record* to be kept in *systematic analysis* is given on p. 88. Blank sheets for the records may be secured at the storeroom. All observations should be noted down *at once*. Particular attention must be paid to recording whether the presence of a trace or of a large or small quantity of a component is indicated.