

CONTENTS

Preface	xi
ONE Fundamentals of Ion Exchange	1
A. Versatility of Ion-Exchange Materials	1
B. The Chemical Nature of Ion Exchangers	2
C. Chemical Formulas for Ion-Exchange Resins	5
D. Physical Properties of Ion-Exchange Resins	6
1. Particle Size and Form	6
2. Swelling and Porosity	8
3. Crosslinkage	8
E. Chemical Properties of Ion-Exchange Resins	9
1. Equivalency of Ion-Exchange Reactions	9
2. Capacity of Exchangers	10
3. Selectivity of the Resins for the Counter-Ion	10
4. Stability	12
F. Mechanisms of Ion-Exchange Reactions	12
1. Ion-Exchange Equilibria	12
2. The Donnan Membrane Theory	13
3. Application of the Law of Chemical Equilibrium to Ion-Exchange Reactions	17
4. The Rate of Ion-Exchange Reactions	22
References	23

TWO Ion-Exchange Processes: Batch and Column Techniques Plate Theory of Chromatography

- A. Definition and Symbols Used in This Chapter**
 - B. Basic Operations**
 - C. Details of the Batch Method**
 - D. The Column Method**
 - 1. General Considerations
 - 2. Frontal Development
 - 3. Displacement Development
 - 4. The Breakthrough Technique
 - 5. Elution Analysis
 - E. The Plate Theory**
 - 1. General Comments
 - 2. The Discontinuous Approach
 - 3. Representation of the Elution Curve as a Gaussian Error Function
 - 4. Methods of Determining the Number of Plates in a Column
 - 5. The Concept of the Plate Theory as a Continuous Process
 - 6. Parameters that Affect the Performance of a Column
- References**

THREE Selecting the Proper Ion-Exchange Material

- A. Preliminary Considerations**
 - 1. Limitation of Ion-Exchange Materials to Four Classes
 - 2. Selecting the Exchanger by a Process of Elimination
 - a. Selection based on the net charge of a solute
 - b. Selection based on the size and net charge of a solute
 - c. Selection based on the chemical and physical environment of a solute
- B. The Ion-Exchange Resins**
 - 1. Trade Names, Manufacturers, and Suppliers
 - 2. Terminology
 - a. Particle size
 - b. Per cent crosslinkage and capacity
 - c. The ionic form of an exchanger
 - d. Formulary
 - 3. Chemical and Physical Stability
- C. Ion-Exchange Celluloses**
 - 1. Fibrous Cellulosic Exchangers
 - 2. Microgranular Cellulosic Exchangers
 - 3. Choice of the Exchanger Type
- D. Ion-Exchange Dextrans and Polyacrylamide Gels**

1. The Gel Matrix	71
2. Preparation	72
3. Some General Characteristics of the Gel-Exchangers	74
4. Selecting the Type of Gel Exchanger	75
E. Inorganic Ion Exchangers	76
1. The Rebirth of Inorganic Ion-Exchange Materials	76
2. Hydrous Oxide Exchangers	77
3. Acid Salt Exchangers	78
4. Heteropoly Acid Salts	79
5. Metal Sulfides	79
6. Inorganic Phosphate Gel	80
F. Miscellaneous Ion-Exchange Materials	80
1. Some Lesser Used Resinous Exchangers	80
2. Pellicular Ion Exchangers	81
G. Ion-Exchange Literature	81
1. Books	81
2. Handbook	82
3. Reviews	83
4. Journals	83
References	84

FOUR Laboratory Columns and Accessories: Operational Techniques 86

A. The Chromatographic Assembly	86
B. Chromatographic Columns	87
1. Column Shape; Length to Diameter Ratio	87
2. Column Types	88
3. Packing the Column	89
a. Multistage batch-packing	93
b. Single-stage batch-packing	93
c. Single-stage pump-packing	93
C. Line Connections from One Accessory to Another	95
D. Apparatus for Delivering the Eluent to the Column	96
E. Detection Devices and Techniques	102
F. Control of Liquid Flow Through the Column Assembly	104
G. Fraction Collectors	105
H. Operation of the Chromatographic Assembly	108
References	109

FIVE Quantitation of Elution Curves 111

A. Analysis of Individual Fractions	111
B. Analysis of Automatically Plotted Elution Curves	112
1. Methods of Obtaining the Area Under Elution Peaks	112

- a. Geometrical integration
- b. Other lesser used methods
- 2. Conversion of Peak Area to Amount of Solute
 - a. Direct relation of peak area to sample composition
 - b. Conversion of peak area into weight or moles of substance through response factors
 - c. Conversion of peak area into quantity of substance through extinction coefficients
- C. Constancy of Operating Conditions
 - 1. Preliminary Considerations
 - 2. Internal Standards
- D. Precision and Accuracy of the Integration Methods
- E. Resolution
- References

SIX Simplification of Some Common Analytical Chemical Operations

- A. Nonchromatographic Operations
- B. Conversion of One Compound to Another
- C. The Standardization of a Salt Solution by Ion Exchange
- D. Removal of Interfering Ions
- E. Preparation of Deionized Water
- F. Purification of Organic Compounds
- G. Determination of Iodate Following Periodate Oxidation of α -Glycol Groups
- H. Catalysis
- I. Recovery of Trace Constituents
 - 1. Concentration of Trace Substances from Water Supplies
 - 2. Recovery of Trace Constituents in General Environmental Work
 - 3. The Isolation of Trace Elements in Food Products
 - 4. Separation of Trace Constituents in the Ore and Metal Industries
 - 5. Recovery of Solutes from Chromatographic Peaks
- References

SEVEN Ion-Exchange Chromatography

Part I Ion-Exchange Chromatography of Organic Substances

- A. Amino Acids, Peptides, and Proteins

1. Amino Acids	145
2. Peptides and Proteins	148
a. Separations on resinous exchangers	148
b. Separation of peptides and proteins on cellulosic ion exchangers	151
B. Separation of Carbohydrate Substances	154
1. Neutral Carbohydrates	154
2. Carbohydrate Derivatives	158
C. Organic Acids	159
1. Introduction	159
2. Detection Methods	159
3. Uronic and Aldonic Acids	160
4. Other Aliphatic Carboxylic Acids	161
5. Separation of Aromatic Acids	162
D. Amino Sugars	164
E. Amines	165
F. Separation of Carbonyl Compounds in the Presence of Bisulfite	168
G. Nucleic Acid Components	168
1. Brief Historical Account	168
2. Purine and Pyrimidine Bases and Nucleosides	171
3. Nucleoside Phosphates	175
4. Polynucleotides	179
5. Nucleic Acids	181
H. Simultaneous Analysis of Different Classes of Compounds	181
 Part II Ion-Exchange Chromatography of Inorganic Substances	 182
A. Emergence of Ion-Exchange Elution Chromatography	182
B. Ion Exchange-Complex Ion Interactions	183
C. Classification and Comparison of Chromatographic Methods	186
D. Separation of Inorganic Cations	186
1. Alkali Metals	186
2. Alkaline Earth Ions	187
3. Transition Elements and Related Metals	193
4. The Rare Earth Elements	195
5. Miscellaneous Metals	197
E. Separation of Inorganic Anions	197
1. Separation of Chloride, Bromide, and Iodide Ions	197
2. Chromatography of Polyphosphate Mixtures	199
3. Separation of Hypophosphite, Phosphite, and Phosphate	200
4. Miscellaneous Other Anions	201
References	201

**EIGHT Utilization of Ion-Exchange Resins for Partition,
Salting-Out, and Ion-Exclusion Chromatography**

- A. Preliminary Considerations
- B. Partition Chromatography of Carbohydrates
- C. Salting-out Chromatography
- D. Separation of Ionic Compounds by Ion Exclusion
and Related Methods
- References

NINE Ligand-Exchange Chromatography

- A. Description of Ligand Exchange
- B. Separation by Ligand-Exchange Chromatography
 - 1. Amphetamine Drugs
 - 2. Purine and Pyrimidine Derivatives
 - 3. Peptides and Amino Acids
- References

Appendix A. Extension of the Donnan Principle

**Appendix B. Determination of the Total Ion-Exchange
Capacity**

**Appendix C. Determination of the Liquid Volume Held
in an Ion-Exchange Column**

Index