

CONTENTS

Introduction	1
SEPARATIONS INVOLVING PHASE CHANGES	
1 Volatilization	5
2 Zone Melting	13
3 Distillation: General Information	21
4 Azeotropic and Extractive Distillations	43
5 Steam and Immiscible Solvents Distillation	49
6 Vacuum Distillation	57
7 Molecular Distillation and Sublimation	71
8 Lyophilization (Freeze Drying)	85
SEPARATIONS INVOLVING EXTRACTION	
9 Extraction: General Concepts	93
10 Continuous Extraction	107
11 Countercurrent (Extraction) Chromatography: The Ito Coil-Planet Centrifugal Extractor	117
12 Solid Phase Extraction	129
13 Supercritical Fluid Extraction	137
SEPARATIONS INVOLVING CHROMATOGRAPHY	
14 Chromatography: General Theory	149
15 Displacement and Multiple Column Partition Chromatography	155
16 Affinity Chromatography	165
17 Size Exclusion Chromatography (Gel Filtration; Gel Permeation)	171
18 Flash Chromatography	179
19 High Performance Liquid Chromatography (HPLC)	183
20 Gas-Liquid Chromatography (GLC)	211
21 Paper Chromatography	249
22 Thin Layer Chromatography	255
SEPARATIONS INVOLVING ION EXCHANGE RESINS	
23 Ion Exchange	269
24 Ion Chromatography	277
25 Ion Retardation, Ion Exclusion, and Ligand Exchange	289

SEPARATIONS INVOLVING ELECTRIC FIELDS

26	Electrodeposition	299
27	Electrophoresis—General	315
28	Immunolectrophoresis	339
29	Polyacrylamide Gel Disc Electrophoresis	345
30	Ion Focusing, SDS, and Two-Dimensional Electrophoresis	351
31	Capillary Zone Electrophoresis	359
32	Field Flow Fractionation	371

SEPARATIONS INVOLVING FLOTATION

33	Purge and Trap and Dynamic Headspace Analysis	385
34	Foam Fractionation, Gas-, and Liquid-Assisted Flotation	395

SEPARATIONS INVOLVING MEMBRANES

35	Osmosis and Reverse Osmosis	413
36	Dialysis and Electrodialysis	423
37	Filtering and Sieving	431

SEPARATIONS INVOLVING MISCELLANEOUS TECHNIQUES

38	Density Gradients	449
39	Centrifugation	455
40	Masking and Sequestering Agents	469
41	Solubility	473
42	Gas Analysis by Portable Orsat	489

APPENDIX A 499

Experiment 1	Volatilization: The Determination of CO ₂ in Either Limestone or Baking Powder	500
Experiment 2	Volatilization: The Determination of Mercury in Hair by Flameless Atomic Absorption Spectroscopy	504
Experiment 3	Zone Refining: The Separation of Traces of Methyl Red from Naphthalene	507
Experiment 4	Azeotropic Distillation: The Production of Absolute Ethanol from 95% Ethanol–Water	509
Experiment 5	Extractive Distillation: Separation of Cyclohexane from Benzene Using Aniline	516
Experiment 6	Steam Distillation: The Separation of Oil of Clove from the Whole Cloves	519
Experiment 7	Immiscible Solvents Distillation: The Use of Toluene to Remove Water from Fruits, Vegetables, and Meats	521
Experiment 8	Vacuum Distillation: A Vacuum Distillation to Purify the Solvent Dimethylformamide	523
Experiment 9	Molecular Distillation: The Determination of Vitamin E (Tocopherol) in Margarine	527
Experiment 10	Sublimation: The Separation of Benzoic Acid from Saccharin	532
Experiment 11	Entrainer Sublimation: The Separation of Caffeine from Coffee by Entrainer Sublimation	535
Experiment 12	Lyophilization: Preparing Freeze-Dried Beverages	538
Experiment 13	Continuous Extraction—Solvent Heavier Than Water: The Determination of Caffeine in Cola Drinks Using a Continuous Extractor; Solvent Heavier Type	540
Experiment 14	Continuous Extraction—Solvent Lighter Than Water: Separating the Anti-amoebic Alkaloids from Ipecac Syrup	544
Experiment 15	Continuous Extraction—The Soxhlet Extractor: Extracting Oil from Nutmeats	548
Experiment 16	Countercurrent Extraction: Separation of the Phenolic Flavor Components, Carvacrol and Thymol, from Oregano Leaves	550
Experiment 17	Solid Phase Extraction: Atrazine, Simazine, and Propazine in Ponds, Lakes, and Rivers (Supelco Applications)	554
Experiment 18	Supercritical Fluid Extraction: The Separation of Oil from Pecans	556

Experiment 19	Displacement Column Chromatography: The Separation of cis-trans-Azobenzene	561
Experiment 20	Multiple Column Partition Chromatography: The Separation of Codeine, Chlorpheniramine, and Phenylephrine in a Cough Syrup	564
Experiment 21	Affinity Chromatography: Separation of a Lectin from Soybeans	567
Experiment 22	Size Exclusion Chromatography: Gel Filtration Chromatography—The Separation of Vitamin B ₁₂ from Dextran by Means of Sephadex G-100	572
Experiment 23	Flash Chromatography: Separation of Vanillin from Vanilla Extract	575
Experiment 24	High Performance Liquid Chromatography: Detecting Explosive Residues	579
Experiment 25	High Performance Liquid Chromatography: Separation of Aspirin, Acetaminophen, and Caffeine in Excedrin	584
Experiment 26	Gas-Liquid Chromatography—Arson Investigation: Detection of Accelerants in Debris by Head Space Analysis	586
Experiment 27	Gas-Liquid Chromatography—Determination of Antioxidants: The Determination of BHA and BHT Antioxidants in Breakfast Cereals	591
Experiment 28	Paper Chromatography: The Determination of Sulfamethazine, Sulfametrazine, and Sulfadiazine in Trisulfa Tablets	595
Experiment 29	Thin Layer Chromatography: Ascending TLC Separations Requiring Color Development—Vasodilator Drugs; the Aliphatic Nitrates	599
Experiment 30	"Instant" Thin Layer Chromatography: The Determination of Antihistamines in Drug Preparations	601
Experiment 31	Two-Dimensional Chromatography: Separation of Sulfamethazine or Carbadox from Medicated Feeds	606
Experiment 32	Ion Exchange: Separations of Several Cations in Mineral Water, Pond Water, or Low Acid Foods	608
Experiment 33	Ion Chromatography: The Separation of Corrosion Inhibitors and Contaminants in Engine Coolants	614
Experiment 34	Ion Retardation: Separating Salt and Urea from Urine	617
Experiment 35	Ion Exclusion: Determination of Total Vitamin C in Applesauce	618
Experiment 36	Ligand Exchange: Determination of Hydrazine, Methylhydrazine, and Dimethylhydrazine in Liquid Rocket Fuels	621
Experiment 37	Electrodeposition: The Determination of Copper and Nickel in a U.S. 5-Cent Piece	623
Experiment 38	Agar Horizontal Strip Electrophoresis: The Separation of the Isoenzymes of Lactic Dehydrogenase (LDH) in Blood Serum	628
Experiment 39	Agarose Gel Horizontal Strip Electrophoresis: DNA Fingerprinting—A Paternity Case	634
Experiment 40	Immuno-electrophoresis: Immuno-electrophoresis of Human Proteins in Urine or Serum	642
Experiment 41	Disc Electrophoresis: Identification of Fish Species by Their Amino Acid Pattern	646
Experiment 42	Ion Focusing Electrophoresis: Ion Focusing of Ferric Leghemoglobins from Soybean Root Nodules	652
Experiment 43	Capillary Zone Electrophoresis: Micellar Capillary Electrophoresis Separation of Aspartame Breakdown Products in Soft Drinks	657
Experiment 44	Field Flow Fractionation: Determination of the Size/Mass of Latex Spheres Using Sedimentation FFF	662
Experiment 45	Purge and Trap: Separation of Aroma and Flavor Compounds in Fresh and Aged Beer	665
Experiment 46	Foam Fractionation: Separation of Detergents from Waste Water	667
Experiment 47	Gas-Assisted Flotation: Separating Suspended Solids from River Water	671
Experiment 48	Liquid-Assisted Flotation—Tomatoes: Separating Fly Eggs and Maggots from Canned Tomatoes	673
Experiment 49	Liquid-Assisted Flotation--Cornmeal and Flour: Separating Insect Fragments and Rodent Hairs from Cornmeal and Flour	676
Experiment 50	Liquid-Assisted Flotation—Leafy Vegetables: Separating Mites, Aphids, and Thrips from Leafy Vegetables	681
Experiment 51	Osmosis: Determining the Osmotic Pressure of a Potato	685
Experiment 52	Dialysis: The Determination of Phosphatase in Milk to Determine Proper Pasteurization	687
Experiment 53	Air Filtration: Separating Pollen and Molds from Air	691
Experiment 54	Density Gradients For Soils: Comparing Soils at a Hit-and-Run Accident	694
Experiment 55	Centrifugation: The Separation of Fat from Milk and Ice Cream	696
Experiment 56	Masking and Sequestering Agents: The Determination of Zn Accelerators in Rubber Products	700

Experiment 57 Solubility of Plastic Materials: Separation and Identification of Thermoplastic Polymers	703
Experiment 58 Solubility of Fibers: Identification of Textile Fibers by Solubility Measurements	704
Experiment 59 Gas Analysis: Separation of CO ₂ , O ₂ , CO, and N ₂ from Car Exhaust Gas Fumes	707

APPENDIX B

Answers to the Review Questions	711
---------------------------------	-----

INDEX	747
--------------	-----