

Contents

Preface — V

Part I: Chemical Laboratory Safety

Chapter 1

Introduction — 3

- 1.1 Safety — 3
- 1.2 Safety Culture — 3
- 1.3 Safety Cycle — 4
- 1.4 Hazard — 4
- 1.5 Incident — 4
- 1.6 Risk — 5
- 1.7 Risk Levels — 5
- 1.8 Risk Evaluation Matrix — 5
- 1.9 Accident — 5
- 1.10 Dangerous Occurrence — 6
- 1.11 Hazardous Substances — 6
- 1.12 Chemicals' Routes of Exposure — 7
- 1.13 Contributing Factors — 7
- 1.14 Nine Principles of Prevention — 7

Chapter 2

General Laboratory Safety — 9

- 2.1 Personal Health — 9
- 2.2 Laboratory Housekeeping — 10
- 2.3 Laboratory Dress — 10
- 2.4 Laboratory Misconduct — 11
- 2.5 Laboratory Visitors — 11
- 2.6 Running Experiments in the Laboratory — 11
- 2.7 Working Alone and After-Hours Work Practices — 11

Chapter 3

WHMIS in the Laboratory — 13

- 3.1 Worker Education — 13
- 3.2 Labeling — 14
- 3.3 Material Safety Data Sheet (MSDS) — 15

Chapter 4

Working with Chemicals — 17

- 4.1 Highly Toxic Chemicals — 17
- 4.2 Flammable and Combustible Liquids — 18
- 4.3 Highly Reactive and Explosive Chemicals — 19
- 4.4 Corrosives — 20
- 4.5 Compressed Gases — 20
- 4.6 Cryogenic Liquids — 21
- 4.7 Transporting Chemicals — 21

Chapter 5

Storage of Chemicals — 22

- 5.1 Chemical Inventory — 22
- 5.2 Storage of Laboratory Chemicals — 22
 - 5.2.1 Acids and Bases — 23
 - 5.2.2 Flammable and Combustible Liquids — 24
 - 5.2.3 Compressed Gases — 24
 - 5.2.4 Ethers and Other Peroxide-Forming Chemicals — 24
 - 5.2.5 Perchloric Acid and Perchlorates — 25
 - 5.2.6 Picric Acid and Nitro Compounds — 26

Chapter 6

Hazardous Chemical Waste Management — 27

- 6.1 Definition of Chemical Waste — 27
- 6.2 Handling and Storage — 28
- 6.3 Labeling Hazardous Waste — 29
- 6.4 Packaging Hazardous Waste — 29
- 6.5 Special Wastes — 29
 - 6.5.1 Solvent Drums — 30
 - 6.5.2 Sharps — 30
- 6.6 Hazardous Waste Pickup — 30
- 6.7 Hazardous Pictograms — 30
- 6.8 The Control of Substances Hazardous to Health (COSHH) — 30
- 6.9 Health Risk Control Measures — 32
 - 6.9.1 Elimination or Substitution — 32
 - 6.9.2 Engineering Control — 33
 - 6.9.3 Administrative Control (Work Habits Control) — 33
 - 6.9.4 Personal Protective Equipment (PPE) — 34

Chapter 7**Hazard Control Measures — 35**

- 7.1 Fume Hoods — 35
- 7.2 Other Laboratory Ventilation — 37
- 7.3 Safe Work Procedures — 37
- 7.4 Personal Protective Equipment — 38
 - 7.4.1 Eye and Face Protection — 38
 - 7.4.2 Gloves and Hand Protection — 38
 - 7.4.3 Laboratory Coats and Aprons — 38
 - 7.4.4 Respiratory Protection — 39
- 7.5 Emergency Showers and Eyewash Stations — 39
- 7.6 Fire Extinguishers — 40

Chapter 8**Fire and Explosion Safety — 41**

- 8.1 Fire Safety — 41
 - 8.1.1 Introduction — 41
 - 8.1.2 Fire Terminologies — 41
 - 8.1.3 Ignition Sources — 41
 - 8.1.4 Makeshift Safety Measures — 42
 - 8.1.5 Precautions — 42
 - 8.1.6 Relative Flammability of Selected Organic Compounds — 42
 - 8.1.7 National Fire Protection Association Classifications — 43
 - 8.1.8 Types of Fire Extinguishers — 43
- 8.2 Explosion Safety — 45
 - 8.2.1 Introduction — 45
 - 8.2.2 Explosive Mixtures — 45
 - 8.2.3 Exothermic Reactions and Explosions — 45

Chapter 9**Laboratory Equipment Safety — 46**

- 9.1 Glassware Safety — 46
- 9.2 Electrical Equipment Safety — 46
- 9.3 Vacuum Pumps and Systems Safety — 47
- 9.4 Heat Sources Safety — 47
 - 9.4.1 Heating Mantles — 47
 - 9.4.2 Oil, Sand, and Salt Baths — 48
 - 9.4.3 Ovens and Furnaces Safety — 48
- 9.5 Refrigerators and Freezers Safety — 48
- 9.6 Decontamination of Laboratory Equipment — 49

Chapter 10

Emergency Procedures — 50

- 10.1 Chemical Spills — 50
- 10.2 Fire and Explosion — 50
- 10.3 Compressed Gas Leaks — 51

Chapter 11

Chemicals and Compatibility Groups — 52

- 11.1 Pyrophoric Chemicals — 52
- 11.2 Oxidizing Agents — 52
- 11.3 Reducing Agents — 53
- 11.4 Water-Reactive Chemicals — 53

Part II: Laboratory Techniques

Chapter 12

Separation Techniques — 57

- 12.1 Filtration — 57
 - 12.1.1 Introduction — 57
 - 12.1.2 Filtration Methods — 58
 - 12.1.3 Filter Media — 58
 - 12.1.4 Filtering Accessories — 59
 - 12.1.5 Operations Associated with the Filtration Process — 60
 - 12.1.6 Gravity Filtration — 61
 - 12.1.7 Vacuum Filtration — 62
- 12.2 Recrystallization — 62
 - 12.2.1 Introduction — 62
 - 12.2.2 Solvent Requirements — 63
 - 12.2.3 Recrystallization of a Solid — 64
- 12.3 Extraction — 66
 - 12.3.1 Introduction — 66
 - 12.3.2 Extraction of Solute Using Immiscible solvents — 66
 - 12.3.3 Extraction Procedures in the Laboratory — 67
 - 12.3.4 Breaking Emulsions — 69
- 12.4 Distillation — 70
 - 12.4.1 Introduction — 70
 - 12.4.2 Simple (Atmospheric) Distillation — 70
 - 12.4.3 Azeotropic Distillation — 72
 - 12.4.4 Fractional Distillation — 72
 - 12.4.5 Vacuum Distillation — 74
 - 12.4.6 Steam Distillation — 74

- 12.5 Refluxing — 75
- 12.6 Evaporation — 77
- 12.7 Rotary Evaporator — 77
- 12.8 Sublimation — 78
- 12.9 Freeze-Drying — 78

Chapter 13

Good Laboratory Practice — 80

- 13.1 Introduction — 80
- 13.2 What Is ISO 9000? — 80
- 13.3 Common Deficiencies at ISO 9000 GLP Audit — 80
- 13.4 Chemical Tracking and Inventory — 81
 - 13.4.1 Barcoding — 82
 - 13.4.2 Barcode Readers — 83
- 13.5 The Laboratory Notebook — 84
- 13.6 Laboratory Statistics — 85
 - 13.6.1 Statistical Terminology — 86
 - 13.6.2 Quality Control — 86
 - 13.6.3 Rejection of Laboratory Data — 87

Chapter 14

Pressure and Vacuum — 88

- 14.1 Introduction — 88
- 14.2 Measuring Atmospheric Pressure — 88
 - 14.2.1 Mercury Barometer — 88
 - 14.2.2 Aneroid Barometer — 88
- 14.3 Gas Laws — 89
- 14.4 Vacuum — 90
 - 14.4.1 Sources of Vacuum in the Laboratory — 90

Chapter 15

Chemicals, Reagents, and Preparation of Solutions — 92

- 15.1 Introduction — 92
- 15.2 Grades of Purity of Chemicals — 92
 - 15.2.1 Commercial or Technical Grade — 92
 - 15.2.2 Practical Grade — 92
 - 15.2.3 USP — 92
 - 15.2.4 CP — 93
 - 15.2.5 Spectroscopic Grade — 93
 - 15.2.6 Chromatography Grade — 93
 - 15.2.7 Reagent Grade — 93
 - 15.2.8 Primary Standard — 93

- 15.3 Common Hazardous Chemicals — **94**
- 15.4 Laboratory Solutions — **94**
- 15.4.1 Chemical Calculations and Computations for Preparation of Laboratory Solutions — **95**
- 15.4.2 Preparation of Standard Laboratory Solutions — **96**

Chapter 16

Determination of Physical and Other Properties — 99

- 16.1 Density — **99**
- 16.1.1 Density of Solids — **99**
- 16.1.2 Density of Liquids — **99**
- 16.1.3 Density of Gases — **101**
- 16.2 Specific Gravity — **101**
- 16.2.1 Pycnometer Method — **101**
- 16.2.2 Hydrometer Method — **102**
- 16.3 Melting Point — **102**
- 16.3.1 Capillary Tube Method — **103**
- 16.3.2 Electric Melting Point Apparatus — **103**
- 16.4 Boiling Point — **104**
- 16.4.1 Boiling Point Determination During Distillation — **104**
- 16.4.2 Test Tube Method — **104**
- 16.4.3 Capillary Tube Method — **104**
- 16.4.4 Electronic Methods — **105**
- 16.5 Viscosity — **106**
- 16.5.1 Newtonian and Non-Newtonian Fluids — **106**
- 16.5.2 Small-Bore Tube Method — **107**
- 16.5.3 Saybolt Viscometer Method — **107**
- 16.5.4 Ostwald Viscometer Method — **107**
- 16.5.5 Viscosity Units and Conversions — **108**
- 16.6 Methods for Determining pH — **109**
- 16.6.1 Colorimetric Determinations: Indicators — **109**
- 16.6.2 pH Test Paper — **109**
- 16.6.3 pH Meter — **109**

Chapter 17

Gravimetric Analysis — 113

- 17.1 Introduction — **113**
- 17.2 Techniques of Representative Sampling — **113**
- 17.2.1 Basic Sampling Rules — **114**
- 17.2.2 Sampling Gases — **114**
- 17.2.3 Sampling Liquids — **115**
- 17.2.4 Sampling Nonhomogeneous Solids — **115**

- 17.2.5 Sampling Metals — 116
- 17.3 Handling Samples in the Laboratory — 117
- 17.3.1 Pretreatment of Samples — 117
- 17.3.2 Crushing and Grinding — 117
- 17.4 Moisture in Samples — 118

Chapter 18

Laboratory Glassware — 119

Chapter 19

Volumetric Analysis — 126

- 19.1 Definitions of Terms — 126
- 19.1.1 Units of Volume — 126
- 19.1.2 Titration — 126
- 19.1.3 Back-Titration — 126
- 19.1.4 Standard Solution — 126
- 19.1.5 Equivalence Point — 127
- 19.1.6 End Point — 127
- 19.2 Typical Physical Changes During Volumetric Analysis — 127
- 19.3 Washing and Cleaning Laboratory Glassware — 127
- 19.3.1 General Rules — 127
- 19.3.2 Cleaning Volumetric Glassware — 127
- 19.3.3 Cleaning Glassware Soiled with Stubborn Films and Residues — 128
- 19.3.4 Ultrasonic Cleaning — 129
- 19.4 Laboratory Glass Drying — 130
- 19.4.1 Drainage Plates and Drainage Shelves — 130
- 19.4.2 Drying Ovens — 130
- 19.4.3 Quick Drying — 130
- 19.4.4 Rinsing Wet Glassware with Acetone — 130
- 19.5 Tools of Volumetric Analysis — 131
- 19.5.1 Volumetric Flasks — 131
- 19.5.2 Pipettes — 131
- 19.5.3 Burettes — 132

Chapter 20

Chromatography — 133

- 20.1 Introduction — 133
- 20.2 Adsorption Chromatography — 133
- 20.3 Partition Chromatography — 133
- 20.4 Thin-Layer Chromatography — 134
- 20.4.1 Introduction — 134
- 20.4.2 Preparation of the Plate for Thin-Layer Chromatography — 135

20.4.3	Thin-Layer Chromatography Procedure —	135
20.5	Gas Chromatography —	136
20.5.1	Introduction —	136
20.5.2	Instrument Design and Components —	136
20.6	Liquid Chromatography —	138
20.6.1	Introduction —	138
20.7	Column Chromatography —	139
20.7.1	Liquid Column Techniques —	140
20.7.2	General Operating Procedure —	140
20.7.3	General Precautions —	142
20.7.4	Sorbents for Column Chromatography —	142
20.7.5	Solvent Mixtures for Use in Column Chromatography —	142
20.8	Ion-Exchange Chromatography —	143
20.8.1	Introduction —	143
20.8.2	Preparing an Ion-Exchange Column —	144

Essential Terms — 147

Abbreviations — 151

Resources and Further Readings — 153

Index — 155