

# CONTENTS

Preface to First Edition	ix
Preface to Second Edition	x
Preface to Third Edition	xi
<b>CHAPTER 1 COMMON PHYSICAL TECHNIQUES USED IN PURIFICATION</b>	<b>1</b>
<b>GENERAL REMARKS</b> .....	<b>1</b>
Safety in the Chemical laboratory.....	3
Trace Impurities in Solvents.....	3
On Cleaning Apparatus.....	4
<b>DISTILLATION</b> .....	<b>5</b>
Techniques.....	5
Distillation at Atmospheric Pressure.....	6
The Distilling Flask.....	6
Types of Column Packings.....	7
Condensers.....	8
Vacuum Distillation.....	9
Vacuum, Schlenk and Glove Box Techniques.....	10
Spinning Band Column.....	10
Steam Distillation.....	11
Azeotropic Distillation.....	11
Isopiestic or Isothermal Distillation.....	12
Sublimation.....	12
<b>CRYSTALLISATION</b> .....	<b>12</b>
Techniques.....	12
Filtration.....	13
Choice of Solvents.....	14
Mixed Solvents.....	15
Solidification from the Melt.....	15
Zone Refining.....	16
<b>DRYING</b> .....	<b>16</b>
Removal of Solvents.....	16
Removal of Water.....	16
Intensity and Capacity of Common Desiccants.....	17
Suitability of Individual Desiccants.....	17
<b>FREEZE-PUMP-THAW and PURGING</b> .....	<b>19</b>
<b>CHROMATOGRAPHY</b> .....	<b>19</b>
Adsorption Chromatography.....	20

Graded Adsorbents and Solvents .....	20
Preparation and Standardisation of Alumina .....	20
Preparation of Other Adsorbents.....	21
Partition Chromatography.....	22
Paired-ion Chromatography.....	22
Flash Chromatography .....	23
Ion-exchange Chromatography.....	23
Ion-exchange Resins.....	23
Ion-exchange Celluloses and Sephadex.....	24
Gel Filtration.....	26
High Performance Liquid Chromatography (HPLC) .....	26
Other Types of Liquid Chromatography.....	26
Vapour Phase Chromatography.....	27
Paper Chromatography.....	29
Thin or Thick Layer Chromatography (tlc) .....	30
<b>SOLVENT EXTRACTION AND DISTRIBUTION .....</b>	<b>31</b>
<b>MOLECULAR SIEVES .....</b>	<b>32</b>
<b>SOME HAZARDS OF CHEMICAL MANIPULATION IN PURIFICATION AND RECOVERY FROM RESIDUES.....</b>	<b>33</b>
Perchlorates and perchloric acid .....	33
Peroxides.....	33
Heavy-metal-containing explosives .....	34
Strong acids .....	34
Solvents.....	34
Salts .....	34
<b>TABLES.....</b>	<b>35</b>
<b>Table 1A:</b> Predicted effect of pressure on boiling point.....	35
<b>Table 1B:</b> Predicted effect of pressure on boiling point.....	36
<b>Table 2:</b> Heating baths.....	37
<b>Table 3:</b> Whatman filter papers.....	37
<b>Table 4:</b> Micro filters .....	38
<b>Table 5:</b> Common solvents used in crystallisation .....	40
<b>Table 6:</b> Pairs of miscible solvents .....	41
<b>Table 7:</b> Materials for cooling baths.....	42
<b>Table 8:</b> Boiling points of gases.....	44
<b>Table 9:</b> Liquids for drying pistols.....	44
<b>Table 10:</b> Vapour pressures of salt solutions .....	45
<b>Table 11:</b> Drying agents for classes of compounds .....	46
<b>Table 12:</b> Graded adsorbents and solvents .....	46
<b>Table 13:</b> Representative ion-exchange resins.....	47
<b>Table 14:</b> Modified fibrous celluloses for ion-exchange.....	48
<b>Table 15:</b> Bead form ion-exchange packings .....	48
<b>Table 16:</b> Columns for HPLC.....	49
<b>Table 17:</b> Liquids for stationary phases in gas chromatography .....	51
<b>Table 18:</b> Immiscible or slightly miscible pairs of solvents .....	51
<b>Table 19:</b> Aqueous buffers .....	52
<b>BIBLIOGRAPHY .....</b>	<b>52</b>

<b>CHAPTER 2. CHEMICAL METHODS USED IN PURIFICATION .....</b>	<b>57</b>
<b>GENERAL REMARKS .....</b>	<b>57</b>
Removal of Traces of Metals from Reagents .....	57
Distillation .....	58
Use of Ion-exchange Resins .....	58
Precipitation .....	58
Extraction .....	58
Complexation .....	59
<b>USE OF METAL HYDRIDES .....</b>	<b>59</b>
Lithium aluminium hydride .....	59
Calcium hydride .....	59
Sodium borohydride .....	59
Potassium borohydride .....	59
<b>PURIFICATION <i>via</i> DERIVATIVES .....</b>	<b>60</b>
Alcohols .....	60
Aldehydes and Ketones .....	60
Amines, as picrates .....	61
as salts .....	61
as <i>N</i> -acetyl derivatives .....	62
as <i>N</i> -tosyl derivatives .....	62
Aromatic hydrocarbons, as adducts .....	62
by sulphonation .....	62
Carboxylic acids, as 4-bromophenacyl esters .....	62
as alkyl esters .....	62
Hydroperoxides .....	63
Ketones, as bisulphite adducts .....	63
as semicarbazones .....	63
Phenols, as benzoates .....	63
as acetates .....	63
Phosphate esters .....	63
Phosphonate esters .....	63
<b>BIBLIOGRAPHY .....</b>	<b>64</b>
<b>CHAPTER 3. PURIFICATION OF ORGANIC CHEMICALS .....</b>	<b>65</b>
<b>CHAPTER 4. PURIFICATION OF INORGANIC AND METAL-ORGANIC CHEMICALS .....</b>	<b>310</b>
<b>CHAPTER 5. GENERAL METHODS FOR THE PURIFICATION OF CLASSES OF COMPOUNDS .....</b>	<b>362</b>
<b>CLASSES OF COMPOUNDS .....</b>	<b>363</b>
Acetals .....	363
Acids, carboxylic .....	363
sulphonic .....	364
sulphinic .....	364

Acid chlorides .....	364
Alcohols, monohydric .....	364
polyhydric.....	365
Aldehydes .....	365
Amides .....	365
Amines .....	365
Amino acids.....	365
Anhydrides.....	366
Carotenoids .....	366
Esters.....	366
Ethers.....	367
Halides .....	367
Hydrocarbons.....	368
Imides.....	369
Imino compounds .....	369
Ketones .....	369
Nitriles.....	369
Nitro compounds .....	369
Phenols.....	370
Quinones.....	370
Salts (organic), with metal ions.....	370
with organic ions.....	370
disulphonates.....	370
Sulphur compounds, disulphides .....	370
sulphones .....	370
sulphoxides .....	371
thioethers .....	371
thiols .....	371
thiolsulphonates (disulphoxides) .....	371
 <b>CHAPTER 6. PURIFICATION OF BIOCHEMICALS AND RELATED PRODUCTS .....</b>	 <b>372</b>
 <b>INDEX .....</b>	 <b>387</b>