

# Contents

Introduction	1
<b>HYDROXYL GROUPS</b>	<b>8</b>
Esterification Procedures, 9	
Acetic Anhydride, 12	
Phthalic Anhydride, 20	
Pyromellitic Dianhydride, 26	
3,5-Dinitrobenzoyl Chloride, 28	
Hydroxyl Groups in Presence of Amino Groups, 38	
Hydroxyl Groups on Adjacent Carbon Atoms (Glycols), 39	
Acidic Hydroxyl Groups (Enols, Phenols, Nitro-Alcohols), 43	
Mixtures of Alcohols, 53	
Phenols only, 54	
Traces of Hydroxy Compounds, 60	
Primary and Secondary Alcohols, 60	
Phenols, 70	
<b>2 CARBONYL GROUPS</b>	<b>73</b>
Oxime Formation, 73	
Bisulfite Addition, 79	
Hydrazone Formation, 85	
Oxidation Methods, 93	
Schiff Base Formation, 114	
Miscellaneous Methods, 118	
Methods for Trace Quantities of Carbonyl Compounds, 124	
Mixtures of Carbonyl Compounds, 129	
<b>3 CARBOXYLIC ACIDS, SALTS, ESTERS, AMIDES, IMIDES, CHLORIDES, AND ANHYDRIDES</b>	<b>130</b>
Carboxylic Acids, 130	
Carboxylic Acid Salts, 133	
Titrimetric Methods, 133	
Combustion Method, 136	

Esters of Carboxylic Acids, 138	
Saponification Methods, 138	
Trace Quantities of Carboxylic Esters, 140	
Carboxylic Acid Amides, 152	
Potentiometric Titrimetric Methods, 153	
Photometric Titration Method, 158	
Reduction Method, 167	
Determination of Primary Amides, 173	
Determination of Trace Quantities of Amides, 174	
Carboxylic Imides, 179	
Carboxylic Acid Chlorides, 179	
Carboxylic Acid Anhydrides, 187	
By Amide Formation, 187	
Traces of Carboxylic Acid Anhydrides, 195	
Determination of Free Acids in Some Anhydrides, 195	
<b>4 ALKOXYL AND OXYALKYLENE GROUPS</b>	<b>203</b>
Alkoxy Groups, 203	
Acidimetric Method, 203	
Iodimetric Approach, 209	
Oxyalkylene Groups, 212	
Iodimetric Method, 212	
In Complex Mixtures, 218	
Determination of Traces of Polyoxyalkylene Compounds, 229	
<b>5 EPOXIDE GROUPS (OXIRANE OXYGEN)</b>	<del>238</del>
Hydrochlorination Methods, 238	
Other Methods, 253	
<b>6 ORGANIC PEROXIDES</b>	<b>255</b>
Iodometric Methods, 255	
Ferrous Thiocyanate Colorimetric Method, 266	
Ferrous-Titanous Reduction Method, 273	
Leuco Methylene Blue Method, 280	
Determination of Peroxides Using Arsenious Oxide, 286	
Colorimetric Method for Trace Peroxide Using N,N Dimethyl- <i>p</i> -phenylenediamine, 288	
<b>7 UNSATURATION</b>	<b>296</b>
Bromination, 297	
Free Bromine in a Solvent, 297	

## Contents

Bromate-Bromide, 301	
Electrically Generated Bromine, 306	
Iodine Number Methods, 313	
Iodine Monochloride, 314	
Iodine Monobromide Method, 316	
Hydrogenation Methods, 318	
Gasometric Methods, 318	
Electrically Generated Hydrogen Method, 333	
Mercuric Acetate Methods, 341	
Determination of Alpha, Beta-Unsaturated Compounds, 350	
Sodium Bisulfite Method, 351	
Morpholine Method, 356	
Acetylenic Unsaturation, 365	
Analysis of Mixtures of Unsaturated Compounds, 371	
8 ACTIVE HYDROGEN	372
Grignard Approach, 372	
Lithium Aluminum Hydride Approach, 379	
9 ACETYLENIC HYDROGEN	381
Silver Methods, 382	
Cuprous Method, 395	
Mercuric Method, 397	
10 ACETALS, KETALS, AND VINYL ETHERS	399
Hydroxylamine Hydrochloride Method, 399	
Bisulfite Method for Acetals and Vinyl Ethers, 400	
Iodimetric Method Specific for Vinyl Ethers, 403	
Mercuric Acetate Method for Vinyl Ethers, 406	
General Method for Traces of Acetals, Ketals, and Vinyl Ethers, 409	
Method for Traces of Acetals of Acetaldehyde and for Traces of Vinyl Ethers, 409	
AMINO GROUPS	417
Titration Methods, 421	
Acylation Methods, 446	
Diazotization and Nitrosation Methods, 446	
Bromination (Aromatic Amines Only), 450	
Coupling (Aromatic Amines Only), 450	

Determination of Amines in Mixtures, 450	
Primary, Secondary, and Tertiary Amine Mixtures, 450	
Primary Amines in the Presence of Secondary and Tertiary Amines, 470	
Secondary Amines in the Presence of Primary and Tertiary Amines, 497	
Tertiary Amines in the Presence of Primary and Secondary Amines, 502	
General Mixtures, 510	
Determination of Trace Quantities of Amines, 510	
12 IMINO GROUPS	
Nonaqueous Titration Methods, 518	
Hydrolysis Methods, 522	
13 TITANOUS AND CHROMOUS REDUCTIONS	526
Titanous Reduction, 526	
Chromous Reduction, 529	
14 HYDRAZINES AND HYDRAZIDES	536
Hydrazines, 536	
Titration Methods, 536	
Oxidation Methods, 537	
Trace Quantities, 542	
Hydrazides, 543	
15 DIAZONIUM SALTS	544
Nitrogen Evolution Measurement, 545	
Coupling Method, 548	
Reduction Method, 549	
Determination of Traces of Diazonium Compounds, 549	
Analysis of Mixtures, 551	
16 QUARTERNARY AMMONIUM COMPOUNDS	
Hydroxides, 552	
Salts, 552	
Trace Quantities of Quaternary Ammonium Salts, 554	

17	ISOCYANATES AND ISOTHIOCYANATES	558
	Primary Amine Method, 558	
	Secondary Amine Methods, 559	
18	MERCAPTANS	563
	Silver Methods, 563	
	Oxidation Methods, 578	
	Mixtures of Mercaptans and Free Sulfur, 582	
	Colorimetric Methods for Trace Quantities, 595	
19	DIALKYL DISULFIDES	600
	Reduction Methods, 600	
	Oxidation Methods, 605	
	Mixtures with Dialkyl Sulfides, 607	
	Mixtures with Mercaptans, 607	
	Determination of Traces, 613	
20	DIALKYL SULFIDES	
21	SULFOXIDES	621
22	SULFONIC ACIDS, SULFONATE SALTS, AND SULFONAMIDES	631
	Sulfonic Acids, 631	
	Sulfonate Salts, 631	
	Sulfonamides, 638	
23	TECHNIQUES AND REASONING IN DEVELOPING NEW ANALYTICAL METHODS OR MODIFYING EXISTING METHODS	645
24	THE ROLE OF QUANTITATIVE FUNCTIONAL GROUP DETERMINATION IN THE IDENTIFICATION OF ORGANIC COMPOUNDS	651
25	USE OF DIFFERENTIAL REACTION RATES TO ANALYZE MIXTURES CONTAINING THE SAME FUNCTIONAL GROUP	
	Mixtures of Hydroxy Compounds, 656	
	Mixtures of Carbonyl Compounds, 662	

Mixtures of Amines, 667

Mixtures of Unsaturated Compounds, 670

Mixtures of Diazonium Compounds, 677

Mixtures of Amides and Nitriles, 679

26 WEIGHING OF VOLATILE OR CORROSIVE LIQUIDS 683

INDEX 687