

# Contents

Introduction	1
<b>HYDROXYL GROUPS</b>	<b>8</b>
Esterification Procedures, 9	
Acetic Anhydride, 12	
Phthalic Anhydride, 22	
Pyromellitic Dianhydride, 28	
3,5-Dinitrobenzoyl Chloride, 31	
Hydroxyl Groups in Presence of Amino Groups, 40	
Hydroxyl Groups on Adjacent Carbon Atoms (Glycols), 42	
Acidic Hydroxyl Groups (Enols, Phenols, Nitro-Alcohols), 46	
Mixtures of Alcohols, 57	
Phenols Only, 57	
Traces of Hydroxy Compounds, 67	
Primary and Secondary Alcohols, 67	
Tertiary Alcohols, 79	
1,2-Diols, 82	
Phenols, 87	
<b>2 CARBONYL GROUPS</b>	<b>95</b>
Oxime Formation, 95	
Bisulfite Addition, 100	
Hydrazone Formation, 107	
Oxidation Methods, 115	
Schiff Base Formation, 138	
Miscellaneous Methods, 143	
Methods for Trace Quantities of Carbonyl Compounds, 148	
<b>3 CARBOXYLIC ACIDS, SALTS, ESTERS, AMIDES, IMIDES, CHLORIDES, AND ANHYDRIDES</b>	<b>161</b>
Carboxylic Acids, 161	
Carboxylic Acid Salts, 165	
Titrimetric Methods, 165	
Combustion Method, 167	

Esters of Carboxylic Acids, 169	
Saponification Methods, 169	
Trace Quantities of Carboxylic Esters, 172	
Carboxylic Acid Amides, 183	
Potentiometric Titrimetric Methods, 184	
Photometric Titration Method, 189	
Reduction Method, 197	
Determination of Primary Amides, 204	
Hypohalite Reaction, 204	
Cyclopropylamides, 216	
Determination of Trace Quantities of Amides, 217	
Carboxylic Imides, 223	
Carboxylic Acid Chlorides, 223	
Carboxylic Acid Anhydrides, 230	
By Amide Formation, 230	
Traces of Carboxylic Acid Anhydrides, 243	
Determination of Free Acids in Some Anhydrides, 243	
Nitriles, 256	
<b>4 ALKOXYL AND OXYALKYLENE GROUPS</b>	<b>263</b>
Alkoxyl Groups, 263	
Acidimetric Method, 263	
Iodimetric Approach, 269	
Oxyalkylene Groups, 278	
Iodimetric Method, 278	
In Complex Mixtures, 283	
Determination of Traces of Polyoxyalkylene Compounds, 294	
<b>5 EPOXIDE GROUPS (OXIRANE OXYGEN)</b>	<b>304</b>
Hydrochlorination Methods, 304	
Other Methods, 319	
Traces of Epoxy Compounds, 322	
<b>6 ORGANIC PEROXIDES</b>	<b>325</b>
Iodometric Methods, 325	
Ferrous Thiocyanate Colorimetric Method, 342	
Ferrous-Titanous Reduction Method, 349	
Leuco Methylene Blue Method, 356	
Determination of Peroxides Using Arsenious Oxide, 362	
Colorimetric Method for Trace Peroxide Using <i>N,N</i> -Dimethyl- <i>p</i> -phenylenediamine, 365	

## UNSATURATION

373

## Bromination, 374

Free Bromine in a Solvent, 374

Bromate-Bromide, 383

Electrically Generated Bromine, 386

## Iodine Number Methods, 394

Iodine Monochloride, 394

Iodine Monobromide, 396

## Hydrogenation Methods, 398

Gasometric Methods, 399

Electrically Generated Hydrogen Method, 413

Hydrogen from Sodium Borohydride, 422

## Mercuric Acetate Methods, 428

## Ozonization, 440

## Epoxidation, 449

Determination of  $\alpha,\beta$ -Unsaturated Compounds, 452

Sodium Bisulfite Method, 452

Morpholine Method, 458

## Acetylenic Unsaturation, 467

## Trace Acetylenes, 473

## 8 ACTIVE HYDROGEN

Grignard Reagent Approach, 479

Lithium Aluminum Hydride Approach, 487

## 9 ACETYLENIC HYDROGEN

Silver Methods, 490

Cuprous Methods, 507

Mercuric Method, 508

## 10 ACETALS, KETALS, AND VINYL ETHERS

510

Hydroxylamine Hydrochloride Method, 510

Bisulfite Method for Acetals and Vinyl Ethers, 511

Iodimetric Method Specific for Vinyl Ethers, 515

Mercuric Acetate Method for Vinyl Ethers, 518

General Method for Traces of Acetals, Ketals, and Vinyl Ethers, 521

Method for Traces of Acetals of Acetaldehyde and for Traces of Vinyl Ethers, 521

	<b>AMINO GROUPS</b>	<b>529</b>
	Titration Methods, 533	
	Acylation Methods, 558	
	Diazotization and Nitrosation Methods, 559	
	Diamines, 563	
	Bromination (Aromatic Amines Only), 565	
	Coupling (Aromatic Amines Only), 566	
	Determination of Amines in Mixtures, 567	
	Primary, Secondary, and Tertiary Amine Mixtures, 567	
	Primary Amines in the Presence of Secondary and Tertiary Amines 586	
	Secondary Amines in the Presence of Primary and Tertiary Amines, 612	
	Tertiary Amines in the Presence of Primary and Secondary Amines, 621	
	General Mixtures, 633	
	Determination of Trace Quantities of Amines, 633	
12	<b>IMINO GROUPS</b>	<b>646</b>
	Nonaqueous Titration Methods, 646	
	Hydrolysis Methods, 650	
13	<b>TITANOUS, CHROMOUS, AND FERROUS REDUCTIONS</b>	<b>654</b>
	Titanous Reduction, 654	
	Chromous Reduction, 658	
	Ferrous Reductions, 664	
14	<b>HYDRAZINES AND HYDRAZIDES</b>	<b>667</b>
	Hydrazines, 667	
	Titration Methods, 667	
	Oxidation Methods, 668	
	Trace Quantities, 673	
	Hvdrazides. 674	
15	<b>DIAZONIUM SALTS</b>	<b>680</b>
	Nitrogen Evolution Measurement, 681	
	Coupling Method, 684	
	Reduction Method. 685	

Determination of Traces of Diazonium Compounds, 685  
Analysis of Mixtures, 687

16 QUATERNARY AMMONIUM COMPOUNDS

Hydroxides, 688

Salts, 688

Trace Quantities of Quaternary Ammonium Salts, 690

ISOCYANATES AND ISOTHIOCYANATES

694

Primary Amine Method, 694

Secondary Amine Methods, 697

Determination of Trace Quantities of Isocyanates, 702

18 MERCAPTANS

Silver Methods, 707

Oxidation Methods, 723

Mixtures of Mercaptans and Free Sulfur, 729

Colorimetric Methods for Trace Quantities, 747

19 DIALKYL DISULFIDES

Reduction Methods, 753

Oxidation Methods, 761

Mixtures with Dialkyl Sulfides, 763

Mixtures with Mercaptans, 763

Determination of Traces, 769

20 SULFIDES

Oxidation, 770

Mixtures of Dialkyl Sulfides and Mercaptans, 775

21 SULFOXIDES

781

Titration method, 781

Titanous Chloride Reduction Method, 782

Dichromate Oxidation, 790

22 SULFONIC ACIDS, SULFONATE SALTS,  
SULFONAMIDES, AND SULFINIC ACIDS

Sulfonic Acids, 792

Sulfonate Salts, 792

## Contents

Sulfonamides, 807

Sulfinic Acids. 812

- 23 TECHNIQUES AND REASONING IN DEVELOPING  
NEW ANALYTICAL METHODS OR MODIFYING  
EXISTING METHODS 815
- 24 THE ROLE OF QUANTITATIVE FUNCTIONAL GROUP  
DETERMINATION IN THE IDENTIFICATION OF  
ORGANIC COMPOUNDS }
- 25 THE USE OF DIFFERENTIAL REACTION RATES  
TO ANALYZE MIXTURES CONTAINING THE  
SAME FUNCTIONAL GROUP 825
- Mixtures of Hydroxy Compounds, 826  
Mixtures of Carbonyl Compounds, 832  
Mixtures of Amines, 837  
Mixtures of Unsaturated Compounds, 840  
Mixtures of Diazonium Compounds, 847  
Mixtures of Amides and Nitriles, 850  
Mixtures of Esters. 852
- 26 WEIGHING OF VOLATILE OR CORROSIVE LIQUIDS
- INDEX