

Contents

Preface	9
Acknowledgements	11
Introduction	12
1. MICROSYNTHESSES ON THIN LAYERS OF SILICA GEL	
Introduction	15
Microsyntheses involving elimination of water	16
Microsynthesis of cyclohexene	16
Microsynthesis of anthraquinone	17
Microsynthesis of cinnamic acid	17
Microsyntheses involving acylations	18
Microsynthesis of phenyl benzoate	18
Microsynthesis of acetophenone	19
Microsynthesis of acetanilide	20
Microsyntheses involving oxidations	20
Microsynthesis of citral	20
Microsynthesis of geranic acid	21
Microsynthesis of heptanoic acid	22
Microsynthesis of di- β -naphthol	22
Microsyntheses involving reductions	23
Microsynthesis of geraniol	23
Microhydrogenation of cyclohexene	24
Microsynthesis involving nitration	24
Micronitration of phenol	24
Microsyntheses involving azo coupling	25
Microsynthesis of azo coupling between α -naphthol and 2,5-dimethoxyaniline	26
Micro-multiple synthesis of azo dyes	28
References	29
Recommended reading	29

2. SEPARATION OF ISOMERS (GEOMETRICAL, OPTICAL) AND OTHER COMPLEXES

Introduction	30
Separation of polyunsaturated fatty acids by argentation TLC and GLC	31
Separation of <i>cis</i> and <i>trans</i> monounsaturated fatty acids by HPLC	32
<i>Cis-trans</i> isomerization of azobenzene	33
Microsynthesis of <i>cis</i> -azobenzene	33
Chromatography of polycyclic aromatic hydrocarbons on impregnated thin-layer plates	34
TLC of carbohydrates in the presence of boric acid	36
Separation of the optical enantiomers of amino acids by reversed-phase HPLC	37
Resolution of amino acid enantiomers by GLC	40
References	42
Recommended reading	42

3. REACTION CHROMATOGRAPHY

Introduction	43
Functional group analysis by pre-column reaction GC	44
Functional group analysis by GC-effluent characterization	46
Determination of epoxide position and configuration by combination reaction TLC/GC	48
Reaction GC in sealed glass capillaries	50
Subtraction GC	52
Ozonolysis-GC of organic compounds in the microgram range	55
Determination of the carbon skeleton and other structural features of organic compounds by GC	58
A thermomicro-procedure for rapid extraction and direct application in TLC (TAS method)	61
References	64
Recommended reading	65

4. DETERMINATION OF FOOD CONSTITUENTS BY CHROMATOGRAPHIC TECHNIQUES

Introduction	66
HPLC determination of sugars in food products	66
TLC of synthetic sweeteners	68
GLC test for honey adulteration by high-fructose corn syrup	69

HPLC determination of theobromine and caffeine in cocoa and chocolate products	70
HPLC analysis of the pungent principles of pepper	71
Determination of tocopherols and tocotrienols in foods and tissues by HPLC	73
Determination of food preservatives	75
HPLC of food preservatives	75
GC of food preservatives	78
TLC of food preservatives	78
TLC of water-soluble vitamins	80
HPLC of antioxidants	81
Reversed-phase HPLC of carotenes in tomatoes	82
TLC of carotenoid pigments in oranges	84
HPLC and TLC of synthetic acid-fast dyes in alcoholic products	86
GLC determination of cholesterol and other sterols in foods	89
HPLC and TLC determination of aflatoxins in corn	91
References	94
Recommended reading	95
5. FORENSIC ANALYSIS	
Introduction	96
Examination of ball-point pen ink constituents by HPLC	96
HPLC determinations of the major alkaloids in <i>Papaver somniferum</i>	97
Separation and identification of cannabis constituents by TLC	99
Ninhydrin as a spray reagent for the detection of some basic drugs on thin-layer chromatograms	101
Identification of post-explosion residues by HPLC and TLC	103
References	105
Recommended reading	106
Subject Index	107

application, has many advantages over other chromatographic techniques and is extensively utilized in this manual.

The success achieved in synthesizing, separating and efficiently identifying minute quantities of compounds by chromatographic methods leads to the solution of problems previously considered insoluble.

The book in its present form includes the following chapters:

1. Microsyntheses on Thin Layers of Silica Gel;
2. Separation of Isomers (Geometrical and Optical);
3. Reaction Chromatography;