CRC

HANDBOOK of CHROMATOGRAPHY

Gunter Zweig
Joseph Sherma
Editors-in-Chief

Pesticides
Volume I

Joanne M. Follweiler
Joseph Sherma

CRC PRESS

CRC Handbook of Chromatography: Pesticides and Related Organic Chemicals

Volume I

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CRC HANDBOOK OF CHROMATOGRAPHY

SERIES PREFACE

The present *Handbook of Chromatography: Pesticides and Related Organic Chemicals,* a joint undertaking by two chemistry professors from Lafayette College, Joanne M. Follweiler and Joseph Sherma, is an outgrowth of the original two volumes published in 1972. In an attempt to update the original data contained in these two volumes, it became apparent that the literature on chromatography in the ensuing 10 years had become too vast to be contained in one or two volumes. Therefore, we decided to attempt to create a series of handbooks which would cover the most important classes of organic and inorganic chemicals in single volumes devoted to one particular class of chemicals. We further attempted to recruit world-renowned experts in the chromatography of particular classes of chemicals. That we have been successful in this endeavor is attested by the volumes already published and other volumes soon to be published. Future volumes of the Handbook of Chromatography will cover steroids, purines and pyrimidines, inorganics, hydrocarbons, and peptides. We invite the users and faithful friends of the Handbooks of Chromatography to suggest additional titles and corresponding candidates for editorship.

The volume on the chromatography of pesticides and related industrial chemicals was initially entitled *Pesticides*. However, it became apparent that this book must include data on compounds such as PCBs, dioxins, and other related compounds that obviously are not pesticides per se, but chromatograph similar to many chlorinated pesticides and often have been confused with pesticides when interpreting chromatographic results. To call these compounds "related industrial chemicals" is, strictly speaking, incorrect, since a number of these compounds have no commercial significance but may occur as contaminants of technical-grade pesticides (e.g., dioxins). However, in the absence of a better description, we hope that the reader will not become confused by the title of this book and will understand our choice of title.

This book should prove to be an invaluable aid and steady laboratory companion to the analytical chemist who is determining pesticide and related industrial chemicals' concentrations in formulations or present as residues in foods, crops, or environmental samples.

Gunter Zweig, Ph.D.
Joseph Sherma, Ph.D.
Winter, 1983/84

INTRODUCTION

The great majority of current methods for the analysis of pesticides and related industrial chemicals, e.g., PCBs, are based on some form of chromatography. Pesticide formulations are often analyzed by gas chromatography (GC) or high performance liquid chromatography (HPLC) using an internal standard compound. Pesticide metabolism studies make wide use of thin layer chromatography (TLC) of radiolabeled compounds. Pesticide residues in foods, biological samples, and environmental samples are usually determined by gas chromatography, high performance liquid chromatography, or thin layer chromatography with densitometric scanning. Sample preparation prior to these determinative steps typically involves solvent extraction of residues followed by purification of the extract by column liquid chromatography (adsorption, partition, gel, or ion exchange). Confirmation of residue identification is often achieved by directly coupled GC/MS or HPLC/MS.

This volume presents a selection of the most important chromatographic data on pesticides and related industrial chemicals published from 1970 through 1980 in the open literature and government manuals. Earlier data on pesticides can be found in Volume I of the *Handbook of Chromatography (General Data and Principles)* (1972) by Zweig and Sherma.

Included in the data tables that make up the major portion of this volume are retention data, chromatographic parameters, detection methods, a summary of sample preparation procedures, and the original literature references. The latter should be consulted for additional details that could not be included in the tables because of the lack of space.

The volume concludes with discussions of detectors and listings of the properties and sources of gas chromatographic liquid phases and supports, HPLC column packings, and thin layer plates. In addition, a list of important books on pesticide analysis and a compound index with common and trade names are given.

The authors have gone to great pains to limit the number of errors in this handbook, but it is inevitable in an undertaking of this complexity that some mistakes will appear. We would appreciate receiving comments or corrections from readers.

THE EDITORS-IN-CHIEF

Gunter Zweig, Ph.D., received his undergraduate and graduate training at the University of Maryland, College Park, where he was awarded the Ph.D. in biochemistry in 1952. Two years following his graduation, Dr. Zweig was affiliated with the late R. J. Block, pioneer in paper chromatography of amino acids. Zweig, Block, and Le Strange wrote one of the first books on paper chromatography which was published in 1952 by Academic Press and went into three editions, the last one authored by Gunter Zweig and Dr. Joe Sherma, the co-Editor-in-Chief of this series. *Paper Chromatography* (1952) was also translated into Russian.

From 1953 till 1957, Dr. Zweig was research biochemist at the C. F. Kettering Foundation, Antioch College, Yellow Springs, Ohio, where he pursued research on the path of carbon and sulfur in plants, using the then newly developed techniques of autoradiography and paper chromatography. From 1957 till 1965, Dr. Zweig served as lecturer and chemist, University of California, Davis and worked on analytical methods for pesticide residues, mainly by chromatographic techniques. In 1965, Dr. Zweig became Director of Life Sciences, Syracuse University Research Corporation, New York (research on environmental pollution), and in 1973 he became Chief, Environmental Fate Branch, Environmental Protection Agency (EPA) in Washington, D.C.

During his government career, Dr. Zweig continued his scientific writing and editing. Among his works are (many in collaboration with Dr. Sherma) the now 11-volume series on *Analytical Methods for Pesticides and Plant Growth Regulators* (published by Academic Press); the pesticide book series for CRC Press; co-editor of *Journal of Toxicology and Environmental Health*; co-author of basic review on paper and thin-layer chromatography for *Analytical Chemistry* from 1968 to 1980; co-author of applied chromatography review on pesticide analysis for *Analytical Chemistry*, beginning in 1981.

Among the scientific honors awarded to Dr. Zweig during his distinguished career are the Wiley Award in 1977, Rothschild Fellowship to the Weizmann Institute in 1963/64; the Bronze Medal by the EPA in 1980.

Dr. Zweig has authored or co-authored over 75 scientific papers on diverse subjects in chromatography and biochemistry, besides being the holder of three U.S. patents.

At the present time (1980/84), Dr. Zweig is Visiting Research Chemist in the School of Public Health, University of California, Richmond, where he is doing research on farmworker safety as related to pesticide exposure.

Joseph Sherma, Ph.D., received a B.S. in Chemistry from Upsala College, East Orange, N.J., in 1955 and a Ph.D. in Analytical Chemistry from Rutgers University in 1958. His thesis research in ion exchange chromatography was under the direction of the late William Rieman, III. Dr. Sherma joined the faculty of Lafayette College in September 1958, and is presently Charles A. Dana Professor of Chemistry in charge of two courses in analytical chemistry. At. Lafayette he has continued research in chromatography and had additionally worked a total of 12 summers in the field with Harold Strain at the Argonne National Laboratory, James Fritz at Iowa State University, Gunter Zweig at Syracuse University Research Corporation, Joseph Touchstone at the Hospital of the University of Pennsylvania, Brian Bidlingmeyer at Waters Associates, and Thomas Beesley at Whatman, Inc., Clifton, N.J.

Dr. Sherma and Dr. Zweig (who is now with the University of California-Richmond) co-authored or co-edited the original Volumes I and II of the *CRC Handbook of Chromatography*, a book on paper chromatography, seven volumes of the series *Analytical Methods for Pesticides and Plant Growth Regulators*, and the Handbooks of Chromatography of drugs, carbohydrates, polymers, and phenols and organic acids. Other books in the pesticide

series and further volumes of the *CRC Handbook of Chromatography* are being edited with Dr. Zweig, and Dr. Sherma has co-authored the handbook on pesticide chromatography. A book on quantitative TLC was edited jointly with Dr. Touchstone, and a general book on TLC was co-authored with Dr. B. Fried. Dr. Sherma has been co-author of eight biennial reviews of column liquid and thin layer chromatography (1968—1982) and the 1981 and 1983 reviews of pesticide analysis for the journal *Analytical Chemistry*.

Dr. Sherma has written major invited chapters and review papers on chromatography and pesticides in *Chromatographic Reviews* (analysis of fungicides), *Advances in Chromatography* (analysis of nonpesticide pollutants), Heftmann's *Chromatography* (chromatography of pesticides), Race's *Laboratory Medicine* (chromatography in clinical analysis), *Food Analysis: Principles and Techniques* (TLC for food analysis), *Treatise on Analytical Chemistry* (paper and thin layer chromatography), *CRC Critical Reviews in Analytical Chemistry* (pesticide residue analysis), *Comprehensive Biochemistry* (flat bed techniques), *Inorganic Chromatographic Analysis* (thin layer chromatography), and *Journal of Liquid Chromatography* (advances in quantitative pesticide TLC). He is editor for residues and elements for the JAOAC.

Dr. Sherma spent 6 months in 1972 on sabbatical leave at the EPA Perrine Primate Laboratory, Perrine, Fla., with Dr. T. M. Shafik, and two additional summers (1975, 1976) at the USDA in Beltsville, Md., with Melvin Getz doing research on pesticide residue analysis methods development. He spent 3 months in 1979 on sabbatical leave with Dr. Touchstone developing clinical analytical methods. A total of more than 230 papers, books, book chapters, and oral presentations concerned with column, paper, and thin layer chromatography of metal ions, plant pigments, and other organic and biological compounds; the chromatographic analysis of pesticides; and the history of chromatography have been authored by Dr. Sherma, many in collaboration with various co-workers and students. His major research area at Lafayette is currently quantitative TLC (densitometry), applied mainly to clinical analysis and pesticide residue and food additive determinations.

Dr. Sherma has written an analytical quality control manual for pesticide analysis under contract with the U.S. EPA and has revised this and the EPA Pesticide Analytical Methods Manual under a 4-year contract jointly with Dr. M. Beroza of the AOAC. Dr. Sherma has also written an instrumental analysis quality assurance manual and other analytical reports for the U.S. Consumer Product Safety Commission, and is currently preparing two manuals on the analysis of food additives for the U.S. FDA, both of these projects also in collaboration with Dr. Beroza of the AOAC.

Dr. Sherma taught the first prototype short course on pesticide analysis with Henry Enos of the EPA for the Center for Professional Advancement. He was editor of the Kontes TLC quarterly newsletter for 6 years and also has taught short courses on TLC for Kontes and the Center for Professional Advancement. He is a consultant for numerous industrial companies and federal agencies on chemical analysis and chromatography and regularly referees papers for analytical journals and research proposals for government agencies. At Lafayette, Dr. Sherma, in addition to analytical chemistry, teaches general chemistry and a course in thin layer chromatography.

Dr. Sherma has received two awards for superior teaching at Lafayette College and the 1979 Distinguished Alumnus Award from Upsala College for outstanding achievements as an educator, researcher, author, and editor. He is a member of the ACS, Sigma Xi, Phi Lambda Upsilon, SAS, and AIC.

THE AUTHORS

Joanne Follweiler has been a full- and part-time member of the chemistry department faculty at Lafayette College for the past 13 years.

She earned a B.S. in chemistry from Muhlenberg College in Allentown, Pa. in 1964. After graduation she joined the information services department with E. I. du Pont de Nemours & Company in Wilmington, Del. She started work in 1966 on an M.S. degree at the University of Pennsylvania. Her thesis was on rearrangements of aromatic sulfur compounds. Besides the normal teaching and fellowship grants, she also worked as an assistant in Penn's Radiocarbon Laboratory. After receiving her Master's degree, she joined the FMC Company in Princeton, N.J. to work in Chemical Information Services as a consultant. In 1968, she left consulting to teach full-time. For 1 year she taught at the Mercer County Community College near Princeton, and in 1969 joined the Lafayette staff. While teaching at Lafayette, she began her doctorate at the University of Pennsylvania. Her dissertation on the structure of some organosulfur complexes was accepted in 1977.

Dr. Follweiler is the author of several articles on organosulfur compounds and has done research in some aspects of this field at Lafayette.

She was elected to the Phi Lambda Upsilon honorary chemical fraternity while in graduate school and has been a member of AAUP.

Dr. Follweiler is married to a chemist, and they are the parents of two daughters.

Joseph Sherma, Ph.D., received a B.S. in Chemistry from Upsala College, East Orange, N.J., in 1955 and a Ph.D. in Analytical Chemistry from Rutgers University in 1958. His thesis research in ion exchange chromatography was under the direction of the late William Rieman, III. Dr. Sherma joined the faculty of Lafayette College in September 1958, and is presently Charles A. Dana Professor of Chemistry in charge of two courses in analytical chemistry. At. Lafayette he has continued research in chromatography and had additionally worked a total of 12 summers in the field with Harold Strain at the Argonne National Laboratory, James Fritz at Iowa State University, Gunter Zweig at Syracuse University Research Corporation, Joseph Touchstone at the Hospital of the University of Pennsylvania, Brian Bidlingmeyer at Waters Associates, and Thomas Beesley at Whatman, Inc., Clifton, N.J.

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Chromatography (advances in quantitative pesticide TLC). He is editor for residues and elements for the JAOAC.

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We also appreciate the typing by Dori Johnson and the checking of parts of the Appendix by Carolyn Zelop.

In addition, we thank the staff of CRC and our coworkers at Lafayette for their support and encouragement.

We are dedicating this book to Dr. Gunter Zweig in honor of his 60th birthday. Dr. Zweig is a pioneer in the chromatographic sciences and the analysis of pesticides. We wish him many more years of active participation in science, technology, and the enterprise of writing and editing.

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Section I
U.S. Food and Drug Administration Gas Chromatographic Data

Introduction

The following table of data on pesticides, metabolites, and related compounds is reprinted from the USFDA *Pesticide Analytical Manual* (PAM), Volume 1, Appendix, 1978.

The table is arranged alphabetically by compound name. The names appear under the column heading **Preferred name.** An asterisk after the name indicates multiple peaks in the gas chromatogram.

The first column in the table is listed as **Std no.** and gives the number the FDA or EPA assigned the pesticide. Numbers 900 to 1000, however, do not signify available standard materials but have been used for computer purposes.

The second column in the table has a heading of **RRT/A.DC-200**; values under this heading are the relative retention times of the corresponding compound relative to aldrin, utilizing a 10% DC-200 (OV-101 can be used interchangeably with DC-200) on 80-100 mesh Chromosorb W HP column packing.

The third column, labeled **RRT/A mixed** is the same as the second except the column packing used to obtain the data was a mixture of 15% QF-1 and 10% DC-200 on 80-100 mesh Chromosorb W HP.

The last two columns in the table are the same as the second and third columns, respectively, except they have the relative retention time listed relative to parathion instead of aldrin. Their headings are **RRT/P.DC-200** and **RRT/P mixed**.

Other chromatographic parameters are

Inlet temperature	225°C
Column temperature	200°C
Carrier gas, flow rate	N_2, 120 mℓ/min
Column length	1.8 m (6 ft)
Column diameter, I.D.	4 mm
Column material	Glass
Detector	Electron capture and thermionic
Temperature of detector	200°C (both detectors)
EC detector voltage (electrometer sensitivity)	DC voltage and electrometer setting at which 1 ng of heptachlor epoxide causes $^1/_2$ full-scale recorder deflection; usually voltage <100 V, sensitivity of 1×10^{-9} or 3×10^{-9} afs
Thermionic detector gas flow rates	Air: 300 mℓ/min; hydrogen-adjusted to give baseline current of 0.2—0.8 $\times 10^{-8}$ amps after flame ignition
Thermionic detector sensitivity	Baseline current (hydrogen flow) adjusted and electrometer sensitivity (1×10^{-8} to 3×10^{-9} afs) set to provide $^1/_2$ fsd for 2 ng of parathion

In the relative retention data, a 0.00 value means no GC response with either detector, while an A designation indicates that a GC response was obtained but retention time was not recorded.

The fourth and fifth columns in the table record sensitivity data, the fourth column with an electron capture detector, and the fifth with a KCl thermionic detector. The data are listed in terms of nanograms required for 50% full-scale deflection ($^1/_2$ fsd) at the standard conditions listed above. A 9999.9 value means that more than 10,000 ng are required for the stated response.

The sixth and seventh columns give data on elution from a Florisil column. The column was prepared by filling a 22-mm (I.D.) chromatographic column with 4 in. (after settling) of activated Florisil topped with about 0.5 in. of anhydrous Na_2SO_4. Petroleum ether (40 to 50 mℓ) was then used to pre-wet the chromatographic column, and a Kuderna Danish concentrator with a calibrated collection tube was the receiver for the eluate. A petroleum ether extract was transferred to the column and allowed to pass through at a rate of not more than 5 mℓ/min. The container was rinsed with two 5-mℓ portions of petroleum ether and the rinsings poured on the column. The walls of the chromatographic column were rinsed with additional small portions of petroleum ether. The column was then eluted (5 mℓ/min) with successive 200-mℓ portions of 6, 15, and 50% of ethyl ether in petroleum ether. The eluates were then concentrated and analyzed by GC. The column in the table with the heading **Mixed ether** refers to elution data from this Florisil column.

The data in the column listed **Methylene chloride** resulted from following the same procedure, but the column was eluted with the following solvent mixtures:

1. 200 mℓ 20% methylene chloride in hexane (v/v)
2. 200 mℓ 50% methylene chloride, 0.35% acetonitrile, 49.65% hexane (v/v)
3. 200 mℓ 50% methylene chloride, 1.5% acetonitrile, 48.5% hexane (v/v)

Data listed in the table under the **Mixed ether** heading are given in terms of which 200-mℓ fraction of eluant the pesticide was recovered in (partial recoveries are included but not so designated). For example, 15 would mean the Florisil column had been eluted with 200 mℓ of 6% ethyl ether-petroleum ether and then 200 mℓ of 15% ethyl ether-petroleum ether, and the pesticide was recovered in the second 200 mℓ — the 15% eluant. A number such as 1550 would mean that the pesticide had been recovered in the 15% plus 50% eluant fractions.

The **Methylene chloride** heading has either 1, 2, or 3 listed, referring to the eluants list above. Mixed numbers are found here also (for example, 23 means the pesticide was recovered in the 50% methylene chloride, 0.35% acetonitrile, 49.65% hexane and also in the 50% methylene chloride, 1.5% acetonitrile, 48.5% hexane eluants).

The designation 0 in both the **Mixed ether** and **Methylene chloride** columns indicates the pesticide was not eluted.

The columns in the table headed with **Thru fat method** and **Thru nonfat method** indicate the sample preparation procedure. The principles of the two methods are listed below.

Fat Method (PAM I 211.1 and 231.1):

> Pesticides and fat are extracted from the product. Three grams of fat are dissolved in petroleum ether, and the residues are partitioned from petroleum ether solution into acetonitrile, which is then diluted with water. Residues are extracted into petroleum ether. By use of Florisil column chromatography, the residues are isolated from coextracted substances. Elution is accomplished with mixed ethyl ether and petroleum ether solutions as designated above. Additional cleanup may be necessary. Residues in concentrated eluates are determined by GC.

Nonfat Method (PAM I 212.1 and 232.1):

> Pesticides are extracted from the sample with acetonitrile or water-acetonitrile. An aliquot of the acetonitrile extract is diluted with water, and pesticide residues are transferred into petroleum ether. Residues are isolated from coextracted substances by chromatography on a Florisil column, eluting with mixed petroleum and ethyl ethers. Additional cleanup may be required for certain products. Residues in concentrated eluates are measured by GC.

Recovery data for these methods is expressed as: C = complete (>80%); P = partial