

CRC

HANDBOOK *of* CHROMATOGRAPHY

Gunter Zweig
Joseph Sherma
Editors-in-Chief

Polymers
Volume I

C. G. Smith	N. E. Skelly
R. A. Solomon	C. D. Chow

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CRC Handbook of Chromatography: Polymers

Volume I

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CRC HANDBOOK OF CHROMATOGRAPHY

SERIES PREFACE

The present volume on the chromatography of synthetic polymers, edited by Charles G. Smith, Richard A. Solomon, Norman E. Skelly, and Carl D. Chow, of the Dow Chemical Company, is an important addition to the series of *Handbooks of Chromatography* begun in 1972 under the aegis of CRC Press. Recently published volumes of this series have covered the subjects of Drugs and Carbohydrates. The analysis and characterization of polymers is greatly aided by various chromatographic methods, and the authors have very ably collected, organized, and edited the most important chromatographic information for polymers, monomers, and oligomers in this volume. This book, like others in the series, consists in the first part of detailed tables of chromatographic data for the important techniques such as size exclusion (gel permeation) chromatography, gas chromatography, thin layer chromatography, and liquid chromatography. The second part of the book is descriptive, giving readers fundamental and practical information on the various techniques especially applicable to polymers and related compounds. This volume is limited to the chromatography of synthetic polymers; natural polymers such as polysaccharides and polynucleotides and nucleic acids are covered elsewhere in the series. As in the past, we ask our faithful readers to inform us of any shortcomings in the book and to call our attention to inadvertent mistakes that invariably go undetected even in the most carefully proofread manuscript.

Gunter Zweig
Joseph Sherma
May, 1982

PREFACE

Volume I of the *Handbook of Chromatography* contains tables of chromatographic data for over 12,000 compounds. The broad scope of these tables includes steroids, drugs, and metal ions in addition to a large number of organic compounds. Volume II presents the theory and practice of gas, liquid-column, thin-layer and paper chromatography. These volumes provide the background for subsequent sections on specific chromatographic applications.

This Volume presents data on the chromatographic analysis of polymers and polymer related compounds. Supplemental theory subsections for gas, liquid-column and thin-layer chromatography describe column and detection systems new to their respective fields since publication of Volume II. This Volume also describes the theory and practice of two specialized techniques for polymer analysis — pyrolysis-gas chromatography and size exclusion chromatography.

This Volume includes tables of chromatographic data abstracted from 1970 to 1980 literature references. Compounds tabulated include residual monomers, plasticizers, additives, antioxidants, and products from pyrolysis of a broad range of polymers. Where applicable, tables include specialized techniques for extraction of additives from the polymer matrix prior to chromatographic analysis.

The Practical Applications subsection provides the reader with a list of commercial suppliers of column packings and packed columns or thin-layer plates.

Finally, the editors wish to acknowledge the contribution of John Cobler who initiated this compilation of polymer chromatographic data 2 years ago but was unable to participate in completion of the manuscript.

Charles G. Smith

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 (1980/81), Dr. Zweig is Visiting Scholar in the School of Public Health, University of California, Berkeley, 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 full professor there in charge of two courses in analytical chemistry. At Lafayette he has continued research in chromatography and has additionally worked a total of 12 summers in the field with Harold Strain at the Argonne National Laboratory, Illinois, James Fritz at Iowa State University, Ames, Gunter Zweig at Syracuse University Research Corporation, New York, Joseph Touchstone at the Hospital of the University of Pennsylvania, Philadelphia, Brian Bidlingmeyer at Waters Associates, Framingham, Mass., and Thomas Beesley at Whatman Inc., Clifton, N.J.

Dr. Sherma and Dr. Zweig [who is now with U.S. Environmental Protection Agency (EPA)] co-authored Volumes I and II of the *CRC Handbook of Chromatography*, a book on paper chromatography, and 6 volumes of the series *Analytical Methods for Pesticides and Plant Growth Regulators*. Other books in the pesticide series and further

volumes of the *CRC Handbook of Chromatography* are being edited with Dr. Zweig, and Dr. Sherma will co-author the handbook on pesticide chromatography. A book on quantitative TLC (published by Wiley-Interscience, New York) was edited jointly with Dr. Touchstone. Dr. Sherma has been co-author of 7 biennial reviews of liquid chromatography (1968 to 1980) and the 1981 review of pesticide analysis for the journal *Analytical Chemistry*. Dr. Sherma has authored major invited chapters and review papers on chromatography and pesticides in *Chromatographic Reviews* (analysis of fungicides), *Advances in Chromatography* (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), and *CRC Critical Reviews in Analytical Chemistry* (pesticide residue analysis). A general book on thin layer chromatography co-authored by Dr. Sherma is now in press at Marcel Dekker.

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 U.S. Department of Agriculture (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 200 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 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 (EPA) jointly with Dr. M. Beroza of the Association of Official Analytical Chemists (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 a manual on the analysis of food additives for the U.S. Food and Drug Administration, 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 is editor of the Kontes TLC quarterly newsletter and also teaches short courses on TLC for Kontes and the Center for Professional Advancement. He is a consultant for several industrial companies and federal agencies on chemical analysis and chromatography and regularly referees papers for analytical journals and research proposals for government agencies.

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 American Chemical Society, Sigma Xi, Phi Lambda Upsilon, Society for Applied Spectroscopy, and the American Institute of Chemists.

THE EDITORS

Charles G. Smith works with the Polymer Analysis Group of the Analytical Laboratories, Michigan Division of the Dow Chemical Company®. He received his B.S. degree from Alleghany College and his M.S. in chemistry from the University of Michigan. Following graduation, he worked three years with the Chemical Division of PPG Industries before joining Dow in 1967.

After 4 years of methods development in the Organic and Agricultural Products Groups within the Analytical Laboratories, Mr. Smith transferred to the Polymer Analysis Group. His expertise over the years has been concentrated on chromatographic separations (gas and liquid) and applications to polymer systems. The last 4 years were spent developing pyrolysis-gas chromatographic capabilities for polymer characterization.

He is chairman of two sections of the American Society for Testing and Materials Committee on analytical method for plastics. Mr. Smith is author or co-author of three publications and numerous Dow® analytical methods.

Dr. Norman E. Skelly has worked on a wide variety of problems in his career at the Dow Chemical Company®. He entered the University of Iowa in 1951 and received his M.S. and Ph.D. degrees in analytical chemistry in 1953 and 1955, respectively. Upon graduation, he joined the Dow Chemical Company®, Michigan Division, where he has the present title of Associate Scientist. His areas of interest include liquid and thin-layer chromatography, electrophoresis and ultraviolet spectrophotometry as they pertain to the solution of organic analytical problems. He is the author or co-author of 25 publications and over 150 Dow® analytical methods.

Carl D. Chow is with the Polymer Analysis Group of the Analytical Laboratories, Michigan Division of the Dow Chemical Company®. He received his B.S. in organic chemistry from the National Taiwan University and his M.S. in polymer chemistry from North Dakota State University. He joined the Physical Research Laboratory of Dow® in 1965 and worked on polymerization kinetics, foam technology, and reinforced plastics. He transferred to the Polymer Analysis Group in 1967. His specialties are molecular weight measurements, gel permeation chromatography (GPC), liquid chromatography, and thermal analysis. He has published in these areas and has lectured on GPC at local ACS sections and universities. He has been one of two review authors for the Application Reviews issue of Analytical Chemistry on Analysis of High Polymers since 1975.

Richard A. Solomon has specialized in the field of gas chromatography during his career at the Dow Chemical Company®. He received his B.S. from Westminster College, New Wilmington, Pa. and his M.S. from Pennsylvania State University in 1955.

He is the author or co-author of five publications and over 200 Dow® analytical methods.

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Section I

Principles and Techniques

1.1 GAS CHROMATOGRAPHY

The basic apparatus and theory of gas chromatography are discussed in *Section A* of the *CRC Handbook Series in Chromatography*.¹ The reader is referred to this same section for details regarding column selection, qualitative and quantitative analysis by conventional gas chromatography. Specific requirements to prepare polymer samples for gas chromatographic analysis (i.e., extraction, derivatization, etc.) are summarized in the tables of Section IV.I in this volume.

Headspace Gas Chromatography

Gas chromatographic headspace analysis is an indirect method for the determination of volatile constituents in liquids or solids by gas chromatographic analysis of the vapor phase which is in thermodynamic equilibrium with the sample in a closed system. Theory and practice of this technique are detailed in books.²⁻³ Kolb⁴ reviews the applications of automated headspace analysis.

In practice, the sample to be analyzed is placed into a glass vial and sealed with a rubber septum. The vial is heated in a constant temperature bath until equilibrium between sample and vapor phase is established for each volatile component. Each component has a unique equilibrium constant or partition coefficient which must be determined experimentally to obtain quantitative analysis. Matrix effects play an important role in determination of these constants. Once equilibrated, a known aliquot of the vapor phase is transferred to the chromatographic column for separation. This transfer is achieved using a gas-tight syringe or, in the case of automatic instruments, an electropneumatic dosing system. With careful calibration, the composition of the vapor phase obtained from the gas chromatogram is related to the volatile content of the original solid or liquid sample. Backflush systems are often employed to eliminate solvent peaks and interferences.

Headspace analysis has achieved considerable importance in the polymer field where the efficient resolution available and the high sensitivity and specificity of gas chromatographic detectors can be exploited. The applications of headspace analysis include the determination of residual solvents in solid chemicals and packaging materials, residual monomers in polymers and olfactory substances in plastics and polymer dispersions.^{5,6} The advantages of this method compared with conventional manipulation of samples are simple sample preparation, minimal analysis time, high sensitivity, and minimal contamination of the column with high boiling or nonvolatile materials.

Detectors

Details of thermal conductivity and flame ionization detector theory are reviewed in an earlier section of this work.¹ The principles of separation for thermionic nitrogen-phosphorus detectors applicable in polymer analysis and other areas are summarized in the following paragraphs.

The thermionic phosphorus-nitrogen detector (PND) is specific for phosphorus and nitrogen containing compounds. An alkali source, connected to negative potential, is situated in the flame of a flame ionization detector. In the presence of phosphorus or nitrogen compounds, a thermionic reaction takes place with the release of negative ions. These are captured by the positive collector electrode giving rise to a detector signal which is supplied to the FID amplifier.

The alkali source consists of a glass bead made of a special rubidium glass fused onto a platinum wire. As the alkali (Rb) is initially present as a nonvolatile rubidium silicate and as conversion to atomic alkali takes place only during the thermionic process, the loss of alkali ions is extremely small, with the result that the detector performance remains constant over a long period of time.

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1.2 PYROLYSIS-GAS CHROMATOGRAPHY

Pyrolysis normally involves the thermal decomposition of organic molecules in the absence of oxygen. Pyrolysis products or fragments generally relate to the structure of the parent compound. Although these decomposition fragments are usually smaller molecules than the parent compound, pyrolysis can result in isomerization or formation of higher molecular weight fragments.

Pyrolysis-gas chromatography (P-GC) combines this thermal decomposition step with gas chromatographic (GC) separation of the pyrolysis fragments. Pyrolysis-gas chromatography-mass spectrometry (P-GC-MS) or direct pyrolysis-mass spectrometry (P-MS) are used to identify pyrolysis fragments. Although theoretically applicable to any organic compound with insufficient volatility for GC separation alone, this text is devoted mostly to P-GC as it applies to polymer analysis. Procedures utilizing mass spectrometric detection are referenced, but details of these detector systems are beyond the scope of this work. Details on the theory and practice of gas chromatography and detectors are described in earlier volumes¹ and Section I.1 of this volume. This section is devoted solely to pyrolysis theory, instrumentation, and applications. Irwin² recently reviewed the field of analytical pyrolysis including a bibliography of review articles and an earlier comprehensive review of the technique was presented by Levy³. Books describing theory and applications of analytical pyrolysis are also available.^{4,5}

Theory

Thermal energy is applied to the polymer sample in the absence of oxygen. For convenience, pyrolysis is frequently performed in the gas chromatographic carrier gas, i.e., helium or nitrogen. The thermal energy applied to the polymeric sample exceeds the molecular bond energies resulting in fragmentation. Mechanisms of fragmentation processes for polymers are reviewed elsewhere.⁶ These fragmentation processes include elimination of small neutral molecules, unzipping the polymer chain yielding monomer units or random chain cleavage. For example, initial decomposition of poly (vinyl chloride) is dominated by loss of HCl. Vinyl polymers such as polystyrene and poly (methyl methacrylate) decompose by unzipping the polymer chain to give high yields of respective monomers. Condensation polymers such as nylon will decompose by a combination of these three mechanisms.

The mechanism of thermal degradation depends on the polymer type, but the nature and relative yields of pyrolysis products are also related to pyrolyzer design and pyrolysis conditions. Critical factors affecting reproducible yield of characteristic pyrolysis fragments include the final pyrolysis temperature, the temperature rise time or rate of heating to this final temperature and sample size. With low final pyrolysis temperatures (300 to 600°C), fragments are generally monomers or other short-chain molecules (oligomers) related to the original polymer structure. As the final pyrolysis temperature increases, yields of these low molecular weight fragments may decrease because of fragmentation to even simpler molecules such as methane, carbon dioxide, and water. Photodiodes and low mass thermocouples are used to accurately measure this final pyrolysis temperature of filament or Curie point pyrolyzers⁷.

The temperature rise time (TRT) is defined as the time interval required to heat the sample from ambient or its initial temperature to the final pyrolysis temperature. If a portion of the polymer sample is pyrolyzed during this heating interval, the duration of heating or TRT has a pronounced effect on the composition and yields of products in the pyrolyzate. In general, rapid rise times are preferred for maximum reproducibility of product yields and minimal side reaction of pyrolysis fragments.

In conjunction with temperature rise time, sample size considerations are important

in determining the composition and yield of polymer fragments. Side reactions are more likely to occur with large samples which cannot be uniformly heated. Generally, microgram quantities are reproducibly pyrolyzed with rise times of 0.3 to 2 sec whereas larger samples require heating times of 5 to 30 sec.

Pyrolyzer Types

Temperature rise time and sample size are frequently dictated by pyrolyzer design. P-GC systems should be designed to minimize dead volume and cold spots while permitting rapid transfer of pyrolysis products to the GC column. Pyrolyzers in use for solids and high-boiling liquids are classified as either pulse-mode or continuous-mode. Curie point and filament pyrolyzers are in the first category and furnace units are operated in the continuous mode. Comparisons of various pyrolyzer types for study of polymer composition are presented in the literature.^{8,9} Table 1 compares critical features of the above mentioned pyrolyzers as well as less common techniques (i.e., laser pyrolysis).

Curie Point

A ferromagnetic wire is heated to a specific temperature (Curie point) using a high-frequency induction coil. The Curie point is defined as the temperature at which the wire alloy becomes paramagnetic and its energy intake decreases. Commercially available pyrolyzers come with wires of alloy compositions to give Curie point temperatures of 300 to 1000°C.¹⁰ A typical Curie point pyrolyzer cross-section is shown in Figure 1. Meuzelaar¹¹ has modified this design to give an automated Curie point pyrolyzer compatible with capillary chromatographic columns.

Factors affecting the TRT and final temperature have been investigated for Curie point pyrolyzers.^{7,12} A major problem affecting reproducibility of Curie point pyrograms appears to be lack of quality control for wire composition resulting in non-reproducible final temperatures. By design, use of Curie point wires prohibits stepped-temperature pyrolysis, i.e., subjecting a sample to a series of increasing temperatures.

Filament or Ribbon

Nichrome and platinum wires in a variety of configurations have been used as pyrolyzer filaments. These filaments can be heated to a final pyrolysis temperature by application of a constant voltage. Filaments heated in this manner have slow, nonreproducible rise times (5 to 25 sec) resulting in poor pyrogram reproducibility. Capacitive boosted filaments are used to give faster rise times (7 to 10 msec) and improved repeatability.⁷

Soluble polymer samples are readily dip-coated from a volatile solvent to ensure uniform sample thickness on the filament. For insoluble polymer samples, coiled filaments are used with and without inserts such as quartz tubes or boats. This technique also increases the TRT because of the mass of the sample holder. Sample size and thickness affect pyrolysis results as previously discussed and these effects have been investigated for filament pyrolyzers.¹³

Ribbon pyrolyzers have more surface area than wire filaments to permit use of larger sample sizes. A commercially available ribbon probe (Chemical Data Systems) operates with a Wheatstone bridge circuit which functions to control the final pyrolysis temperature and TRT. This unit gives typical TRT of 10 msec to 600°C for microgram samples applied as thin films from solution. A V-groove ribbon is available to hold solid, insoluble polymer samples.

Furnace

Continuous mode furnace pyrolyzers consist of a pyrolysis tube or chamber, a sam-

Table 1
COMPARISON OF PYROLIZERS*

Characteristic Method	Sample size	Sample form requirements and limitations	Temperature range	Pyrolysis element temperature control	Catalytic reactions within pyrolysis chamber	Temperature rise time to 600°C	Repeatability	Disadvantages	Advantages
Boat	1—5000 μ g	All forms 1. Liquids 2. Solids	Up to 1500°C	Continuously variable	Slight	20—50 sec	Fair	1. Slow rise time 2. Requiring large samples 3. Does not lend itself to capillary columns	1. Any material, liquid or solid 2. Widest temperature range 3. Wide sample size range 4. Can be purchased
Conventional filament and ribbon	10—1000 μ g	1. Soluble in a solvent	Up to 1200°C	Continuously variable	Yes	5—25 sec	Fair	1. Slow rise time 2. Aging of metal 3. Solid material hard to apply	1. Most widely used 2. Wide temperature range 3. Purchase or built sample 4. Simple to apply
Capacitive boosted filament	5—10 μ g	1. Soluble in a solvent	Up to 900°C	Continuously variable	No	7—10 msec	Good	1. Solid material must be soluble 2. Must use small sample size 3. Cannot be purchased	1. Fast rise time 2. Wide temperature range 3. Good repeatability
Curie point low power (30—100 W)	10—50 μ g	1. Soluble in a solvent 2. Some crystals and powders	Up to 980°C	Limited to 356, 480, 510, 600, 770, 980°C	Yes	1.5 sec	Good	1. Solid material hard to apply 2. Pyrolysis chamber must be heated 3. Slow rise time	1. Accurate temperature 2. Good repeatability 3. Can be purchased
Curie point high power (> 1000 W)	10—50 μ g	1. Soluble in a solvent 2. Some crystals and powders	Up to 980°C	Limited to 356, 480, 510, 600, 770, 980°C	Yes	70 msec		1. Solid material hard to apply 2. Pyrolysis chamber must be heated	1. Fast rise time 2. Accurate temperature 3. Good repeatability 4. Can be purchased