TECHNIQUES OF CHEMISTRY

VOLUME XVI

# SEPARATIONS BY CENTRIFUGAL PHENOMENA

HSIEN-WEN HSU

*The University of Tennessee*

EDITOR:

EDMOND S. PERRY

TECHNIQUES OF CHEMISTRY

VOLUME XVI

# SEPARATIONS BY CENTRIFUGAL PHENOMENA

HSIEN-WEN HSU

*The University of Tennessee*

*EDITOR:*

EDMOND S. PERRY

Research Laboratories
Eastman Kodak Company
Rochester, New York

A WILEY-INTERSCIENCE PUBLICATION

JOHN WILEY & SONS

New York • Chichester • Brisbane • Toronto

***Library of Congress Cataloging in Publication Data:***

Hsu, Hsien-Wen, 1928-
Separations by centrifugal phenomena.

(Techniques of chemistry; v. 16)
"A Wiley-Interscience publication."
Includes index.
1. Centrifugation. 2. Separation (Technology)
I. Perry, Edmond S. II. Title. III. Series.
QD61.T4 vol. 16 [QD54.C4] 542s [543'.083] 81-4991
ISBN 0-471-05564-6 AACR2

Printed in the United States of America

10 9 8 7 6 5 4 3 2 1

# INTRODUCTION TO THE SERIES

Techniques of Chemistry is the successor to the Technique of Organic Chemistry Series and its companion—Technique of Inorganic Chemistry. Because many of the methods are employed in all branches of chemical science, the division into techniques for organic and inorganic chemistry has become increasingly artificial. Accordingly, the new series reflects the wider application of techniques, and the component volumes for the most part provide complete treatments of the methods covered. Volumes in which limited areas of application are discussed can be easily recognized by their titles.

Like its predecessors, the series is devoted to a comprehensive presentation of the respective techniques. The authors give the theoretical background for an understanding of the various methods and operations and describe the techniques and tools, their modifications, their merits and limitations, and their handling. It is hoped that the series will contribute to a better understanding and a more rational and effective application of the respective techniques.

Authors and editors hope that readers will find the volumes in this series useful and will communicate to them any criticisms and suggestions for improvements.

The editor of the volume and I gratefully acknowledge the assistance of Professor Howard Brenner of the University of Rochester in the editorial work.

ARNOLD WEISSBERGER

*Research Laboratories*
*Eastman Kodak Company*
*Rochester, New York*

# PREFACE

Separations using centrifugal phenomena have been widely used in laboratories and industries for many years. Such methods grew from nineteenth century farm-oriented milk separation to a fundamental technique for determining molecular properties of either natural or synthetic polymers in laboratories and to a basic unit operation component in modern industries today.

Over the last twenty-five years, important contributions to the general understanding of centrifugal separation and ultracentrifugal analysis have appeared in the literature. These contributions are both theoretical and experimental, covering widely diversified fields. Very little effort seems to have been made toward the aims of organizing all this new information in a coherent fashion. The scientific information is scattered in a variety of technical publications; keeping track of it is a difficult task, which is not made simpler by the fact that even the nomenclature has not been standardized.

The objective of writing this book has been to organize this technical information in as coherent a way as possible while emphasizing uniqueness in methodology and pertinent theory for various kinds of centrifugations. In this attempt a framework of transport phenomena to bring together some of the principles of centrifugal separations has been used.

The topics in the book are arranged according to gas, liquid, and solid phases, in the order of increasing phase densities. Much space has been devoted to liquid centrifugation because it is now one of the most basic and valuable techniques in chemical and biological laboratories. The proper application of many centrifugation techniques requires considerable expertise. It was realized that many of the pertinent details scattered in the literature for the laboratory techniques had to be drawn together first, so that the laboratory researchers as well as the novice would benefit from these modern technologies. In addition, many separational and characterizational examples are illustrated in details. It is hoped that this volume complements those in other volumes in the *Techniques of Chemistry* series.

Many of my former colleagues of the Molecular Anatomy (MAN) Program at Oak Ridge National Laboratory have contributed either directly or in-

directly to the present treatise. I acknowledge gratefully the contributions of the following: Dr. J. W. Holleman, who read the entire draft and made numerous corrections and suggestions and whose friendship provided valuable encouragement; Messrs. J. N. Brantley, R. E. Canning, L. H. Elrod, W. W. Harris, L. C. Patrick, C. T. Rankin, Jr., and D. D. Wills, for their assistance and advice; the sharing of their extensive knowledge has been invaluable to me in many respects; Dr. N. G. Anderson, under whose leadership zonal centrifuges have been developed and who provided a scholarly atmosphere when I worked at the MAN Program on a part-time basis from October 1967 to January 1973.

I also wish to acknowledge the generous support and encouragement of the Department of Chemical, Metallurgical and Polymer Engineering of the University of Tennessee-Knoxville in providing the clerical staff and supplies for the preparation of the manuscript; Dr. H. F. Johnson, who gave constant encouragement and support in the preparation of the manuscript; Dr. E. E. Stansbury, who gave many constructive suggestions; Mrs. Kay Davis, Mrs. Sally O'Connor, and Mrs. Janet Deurlein, who typed the original and revised manuscripts; and Mr. T. H. DeHart, who helped in many photographic problems.

In writing a book such as this, it was necessary to use some data from the manuals of manufacturers and materials from many publications. I thank the following for giving permission to use their publications: Academic Press, Inc.; American Chemical Society; Americal Elsevier Publishing Co.; American Institute of Chemical Engineers; American Instrument Co.; American Society for Microbiology; Beckman Instruments Co., Spinco Division; British Biochemical Society; Butterworth Publishers, Inc.; Cambridge University Press; Department of Energy, Electro Nucleonics, Inc.; Instrumentation Specialities Co.; John Wiley & Sons, Inc.; Marcel Dekker, Inc.; McGraw-Hill Book Co.; Oak Ridge National Laboratory; The Rockefeller University Press; Union Carbide Corp.; W. B. Saunders Company; and The Wistar Institute of Anatomy and Biology.

I also thank Dr. R. K. Genung, Oak Ridge National Laboratory, who on many occasions has assisted me in the preparation of manuscripts; and finally, my wife Cecilia, who provided a comfortable atmosphere and also shared with me all the burdens that devolve upon an author's wife.

HSIEN-WEN HSU

*Knoxville, Tennessee*
*June 1981*

**TECHNIQUES OF CHEMISTRY**

ARNOLD WEISSBERGER, *Editor*

---

VOLUME XVI

# CONTENTS

## CHAPTER I



## Chapter II

## Chapter III



## CHAPTER IV

**CHAPTER V**

## CHAPTER VI

## CHAPTER VII



## APPENDIX A

Chapter I

# INTRODUCTION

Separation by centrifugal phenomena is based on the transfer of materials from one phase to another by mechanical means utilizing differences in particle density and size in mixtures under an applied centrifugal force field.

Separation of immiscible liquids and insoluble particles has been occurring in nature since the beginning of the universe. The conscious application of centrifugal force to aid separation, however, is recent. Centrifugal separators began finding applications in the process industries as late as about 100 years ago. The earliest uses were in sugar manufacture and in separating cream from milk. The first continuous centrifugal separator was probably invented in 1877 by a Swedish engineer, Dr. Carl Gustaf Patrik DeLaval, in order to separate cream from milk. This hand-cranked farm tool was disclosed in an English patent in 1878 and a corresponding U.S. patent in 1881 [1]. The applications in the sugar and the dairy industries still account for a substantial number of the new separators made each year, but the number of other applications is constantly increasing. Types of centrifugal separators also proliferated from the original two to a wide diversity of machines.

Industrial centrifuges underwent rapid development during World War II, notably in the field of isotope separation or enrichment by gas centrifuges. A pilot plant-scale ultracentrifuge to separate $^{235}U$ by centrifugal force has been developed [2]. Centrifuges operated by remote control have been used in the isolation of plutonium [2]. The preparation of blood plasma, the concentration of rubber latex, and the separation of penicillin solvents are only a few of many other new uses of the centrifugal method.

The recent population explosion has presented us with various new problems, for example, water pollution, industrial wastes, shortage of resources, and so forth. Centrifuge separators have also been successfully applied to these new problems. For instance, centrifuges are used to purify and recover oils, coolants, and hydraulic fluids in factories, thus saving industry hundreds of thousands of dollars and helping conserve our diminishing resource of energy-providing petroleum. Using centrifuges in recycling operations minimizes handling, hauling, and disposal costs for used lubricants. In the oil industry, centrifuge separators recover otherwise wasted "slop" oil. Centrifuges are used on board ship and on oil platforms to remove salt water and contaminants from fuels and lubricating oils.

In the food industries, centrifuges are used in many operations, for example, in the centrifugal extraction of edible proteins from both animal and vegetable products. In wineries and breweries, centrifugal separators clarify the liquids by removing yeast and other solid matter. Centrifuges, many with programmable controls, are increasingly used to process pharmaceutical preparations. Impurities in coffee, tea, and fruit juices, for example, citrus juices, are removed with the aid of centrifuges. In pulp and paper mills, centrifuges help in producing tall oil, which in turn is used in the production of coatings and soaps and in many chemical processes. To help agricultural producers in their efforts to keep pace with the world's food requirements, centrifuges dewater organic liquids that are used in the production of fertilizers. Industrial and municipal wastes are treated with the help of centrifuges to dewater sludge, thus aiding in the preservation of water resources. In mining operations, centrifuges recover minerals, such as uranium, with the aid of a new liquid ion-exchange method. In steel mills, centrifuges reclaim and recycle lubricating oils and thus play an important role in a manufacturing process that is basic to nearly every industry. Centrifuges are also used in the production of television tubes to recover rare earth phosphors that might be lost as waste.

In cheese plants, centrifuges separate small curd fines from whey to recover cheese that would otherwise be lost. Silver halide coatings used in photographic film and paper are recovered from wash water and put back into production with the aid of centrifuges. Thus, centrifuges are tools with a thousand uses.

Common industrial centrifugal separators handle tons of material at a moderate cost. The current operating conditions of these separators are as follows: temperatures from $-62$ to 230°C; absolute pressures from 5 mm Hg to about 1000 kPa; particles sizes from to 1 $\mu$ in diameter or less up to 0.65 cm.

Centrifuges are not, of course, the answer to all separation problems. In liquid–liquid separations, the specific gravities of the phases must normally differ by a minimum of 3%. In removing solids, absolute clarity of the effluent liquor can rarely be expected. Often, 1 to 5% of the solids fed to the machine remain in the discharged liquid. Continuous and automatic filtering centrifuges are limited to relatively coarse, free-filtering solids.

Advances in centrifuge technology are not limited to large-scale industrial centrifuges. Small centrifuges are also used extensively in laboratories for research and development. Since the late Professor T. Svedberg began to apply centrifugal force for the study of colloid systems in the 1920s, progress in laboratory centrifugations has continued in two directions. The first line of development has produced the analytical ultracentrifuge, which incorporates an optical system for the analysis of a small amount of material which is being centrifuged through a homogeneous medium (the solvent) under ideal conditions. The analytical centrifuge is now widely used not only to characterize molecular weight but also to give insight into the size, shape, density, and the base composition and activity of biopolymers and other macromolecules. The second line of development has produced the preparative centrifuge, which uses centrifugal force to sediment the solid phase of materials. The preparative centrifuge is a simpler instrument and does not have an optical system. However, recent rapid technical progress and development permit the preparative centrifuge not only to completely separate several or all components in a mixture but also to perform analytical measurements. This versatile technique is known as density-gradient centrifugation.

The density gradient method involves a supporting column of fluid whose density increases from top to bottom of the centrifugal bottle. The extreme usefulness of centrifugal systems with swing-out buckets in density-gradient centrifugal separation has been well proved. However, the relatively small volumes that can be handled in high centrifugal fields, together with the mechanical handling procedures, impose certain limitations on the technique and the resolution. To eliminate these limitations, N. G. Anderson and his coworkers at Oak Ridge National Laboratory developed a series of density-gradient centrifuge rotors for the mass separation of subcellular particles including viruses. These centrifuges are known as "zonal centrifuges" [3].

## 1 CLASSIFICATION AND APPLICATION

Industrial centrifuges may be classified in several ways, but almost all of them fall into one of two classes. In one class are the machines that separate by sedimentation and depend on the difference in density between phases. The other class contains filtration machines, in which liquid is forced through a filter medium by centrifugal action. The former is generally classified as a *centrifugal settling machine*, the latter as a *centrifugal filter*. A few machines perform both functions in a single unit. Each class may be subdivided into various types, depending on the configuration of the machines.

The centrifugal settling machine may be further subclassified into three types, each with a broad field of application. These are (1) tubular type, (2) disk type, and (3) decanter. The centrifugal filter may be subclassified into (1) basket type, (2) push type, (3) screw-conveyer type, and (4) self-discharge type. In each type are machines that discharge solids intermittently or continuously.

### Centrifugal Settling Machines

This class contains high-speed, high-force separators with manual removal of accumulated solids; high-speed separators with continuous or periodic discharge of a slurry or sludge; moderate-speed units with continuous discharge of sludge; and slow-speed, large-diameter separators with intermittent solids removal.

High-speed sedimentation centrifuges with manual solids discharge include tubular and disk machines. Their uses include liquid–liquid separations, clarification of liquids by the removal of small amounts of solids, concentration of emulsions, classification of fine solids, and the partial separation of gases of differing molecular weight.

Industrial tubular settling centrifuges measure approximately 10 to 15 cm (4 to 6 in.) in diameter, rotate at speeds up to 15,000 rpm, and generate centrifugal forces as high as 16,000 *g*. They are simple in construction, easy to clean, and have the best operating characteristics of any centrifuge on viscous liquids. Typical applications include purification of lubricating and fuel oils; harvesting of bacteria; clarification of nitrocellulose dope and molten chicle; dewaxing of petroleum residual stocks in hydrocarbon solution; and recovery of finely divided metal particles such as silver from film scrap, platinum from spent catalyst, and germanium from forming and sawing operations. Throughput normally ranges from 4 to 40 liters (1 to 10 gallons) of liquid per minute. A modified tubular settling centrifuge containing several concentric annular chambers connected in series is used for relatively simple clarification problems. Its chief application is in clarifying brewer's wort at rates up to about 120 liters (30 gallons) per minute. A sketch of a tubular settling centrifuge is presented in Fig. 1.1*a*.

The solid-wall disk centrifuge was first developed as a milk separator and still finds wide application in this service. It is also useful for concentrating other emulsions, such as natural and synthetic rubber latexes. It finds a wide variety of applications that overlap the applications of tubular machines to some extent. Typical uses include the purification of lubricating and fuel oils; separation of wash water from fats in refining vegetable oils, fish oils, and whale oil; separation of acid sludge from the acid treatment of petroleum stocks; separation of solvent extracts from fermentation broths; and removal of water from jet fuel. Industrial disk settling centrifuge separators range from 15 to 76 cm (6 to 30 in.) in inside diameter, with liquid throughputs as great as several hundred gallons per minute on easy separations. Some disk centrifuges include openings in the bowl wall for periodic discharge of accumulated solids. These openings may be individually valved ports that are self-actuated by the accumulation of solids or externally actuated from a hydraulic circuit, or they may be peripheral slots which are automatically uncovered at intervals by hydraulic action. Self-actuating valve bowls are used in recovering wool grease from scouring liquors. Externally actuated valve bowls find application when the flow rate required for satisfactory clarification is low and the density of the separated sludge is not much greater than that of the liquid. This type of settling machine is used to remove excess pulp from pineapple and orange juices.