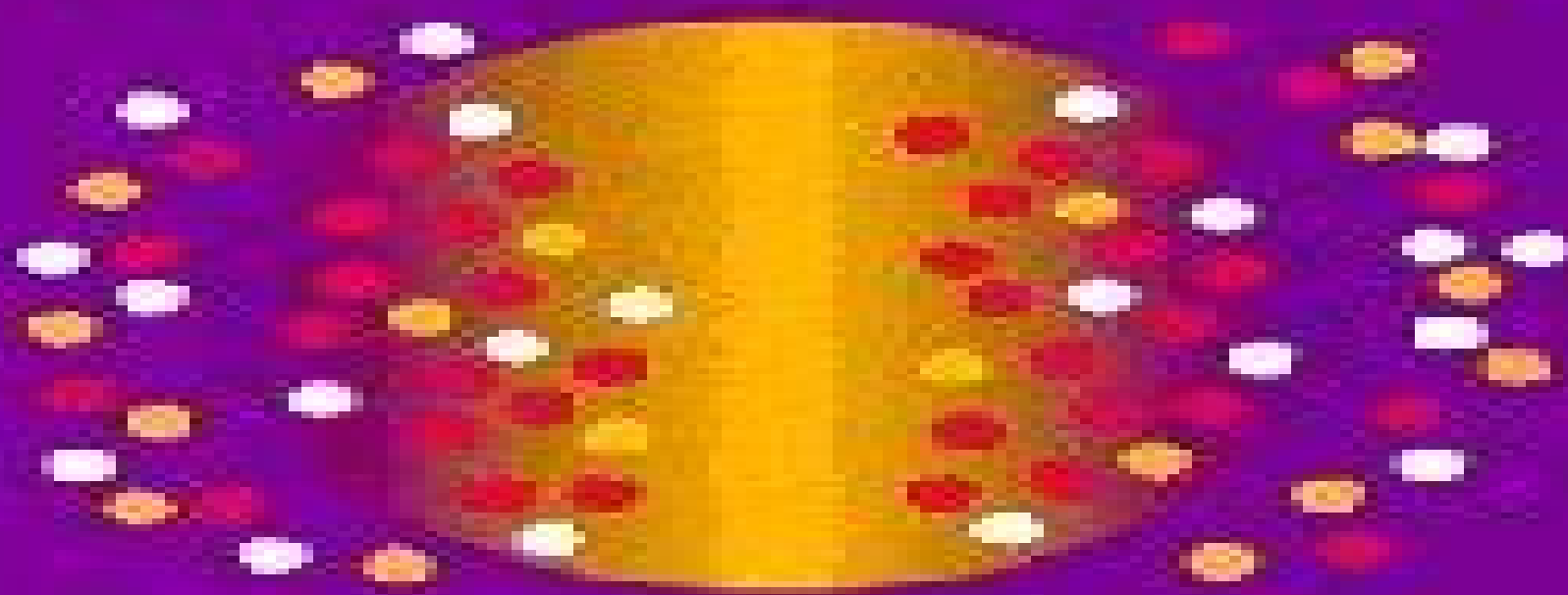


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Hochauflösende Online-NMR-Spektroskopie für das Reaktions- und Prozeßmonitoring

Beispiele zur Anwendung in der Verfahrenstechnik Michael Maiwald, 2012-06-15 Zusammenfassung Die moderne Reaktions- und Trenntechnik in der chemischen Industrie ist eine Hochtechnologie. Die dort eingesetzten Verfahren zur Reaktionsführung und Auftrennung der Produkte sind weitestgehend optimiert. Zu diesem Stand hat die instrumentelle Analytik, speziell die Prozeßanalytik, wesentlich beigetragen. Das Reaktions- und Prozeßmonitoring ist eine wichtige Hilfe zum Verständnis der komplexen Zusammenhänge. In der vorliegenden Arbeit wird der Einsatz der NMR-Spektroskopie in verfahrenstechnischen Anwendungen wie dem Reaktions- und Prozeßmonitoring diskutiert und alle dazu notwendigen Grundlagen erläutert. Solche Anwendungen erfordern häufig Techniken, mit denen hochaufgelöste Spektren zerstörungsfrei aufgenommen werden können, oft bei erhöhtem Druck und erhöhter Temperatur. Neben der quantitativen Bestimmung der Zusammensetzung komplexer reagierender Mischungen besteht gleichzeitig die Möglichkeit zur Identifikation von Nebenprodukten. Dieses gelingt insbesondere durch die Nutzung der NMR-Spektroskopie als Online-Methode, die in der Literatur bislang nur in wenigen Einzelfällen beschrieben und trotz ihrer enormen Möglichkeiten noch nicht konsequent angewendet wird. Durch die fortschreitende Entwicklung auf dem Gebiet der NMR-Spektroskopie kann eine Online-Anbindung heute durchgehend mit Hilfe kommerziell erhältlicher Komponenten erfolgen, wie die vorliegende Arbeit zeigt. Fallende Kosten in der Beschaffung und dem Betrieb leistungsfähiger NMR-Spektrometer machen die Methode auch im verfahrenstechnischen Umfeld mittlerweile sehr attraktiv. Die Attraktivität der Methode gewinnt neuerdings insbesondere durch die erweiterten Einsatzmöglichkeiten kompakterer NMR-Magneten mit geringen Streufeldern. Obwohl sich in den Ingenieurwissenschaften damit vielfältige Einsatzmöglichkeiten ergeben, wird die NMR-Spektroskopie dort bislang noch leider kaum genutzt. Besondere Herausforderungen stellen sich u.a. dadurch, daß sich weder deuterierten Komponenten einsetzen lassen, noch die Probe in einer geeigneten Weise in einer Probenvorbereitung verändert werden kann. Damit kommen hochkonzentrierte Mischungen zur direkten Untersuchung. Dieses hat signifikante Rückwirkungen auf die einzusetzende NMR-Methodik, die im Rahmen der Arbeit umfassend untersucht wurde. In der Regel lassen sich die Probleme bei entsprechender experimenteller Vorgehensweise umgehen. Zur Garantie quantitativer Messwerte wurden alle eingesetzten Mess- und Auswertungsstrategien ausführlich untersucht und teilweise erweitert. Um quantitativ aussagekräftige Online-NMR-Spektren mit Hilfe der ^1H und ^{13}C -NMR-Spektroskopie von technischen Mischungen zu erhalten, wurden folgende Aufgaben gestellt: Entwicklung und Validierung von Vorgehensweisen zur Messung hochkonzentrierter technischer Mischungen ohne Probenvorbereitung und ohne Zusatz deuterierter Komponenten; Konstruktion geeigneter Apparaturen zur Untersuchung von Reaktionsgleichgewichten und Kinetiken für verschiedene Druck- und Temperaturbereiche, die sich optimal für die Online-NMR

Spektroskopie einsetzen lassen Ankopplung der NMR Spektrometers an die unterschiedlichen Apparate mit Hilfe von NMR Durchflüssen Schaffung einer möglichst noninvasiven Untersuchungsmethode hinsichtlich aller Probenparameter z.B. Druck Temperatur Schaffung und Erprobung von Mess- und Auswertungsstrategien im Hinblick auf quantitative Parameter Erweiterung des zugänglichen Druck- und Temperaturbereiches der Messungen sowie Verknüpfung des Zeitfensters für Messungen durch geeignete Peripherie und konstruktiven Veränderungen am NMR Probenkopf Als Beispiel werden Messungen an binären und ternären flüssigen Mischungen aus Formaldehyd Wasser und Methanol diskutiert In diesen Systemen ist Formaldehyd fast ausschließlich in Polyoxymethylen Glykolen und hemiformalen chemisch gebunden Die chemischen Reaktionen in formaldehydhaltigen Mischungen bestimmen deren thermodynamisches Verhalten sowie ihre Trennung mit thermischen Verfahren Die NMR Spektroskopie ist das zentrale analytische Verfahren mit dem sich diese Vorgänge aufklären und quantifizieren lassen Für die hier vorgestellten Untersuchungen zum Prozessmonitoring kamen auch eine Online NMR-Kopplung mit einem Dünnschichtverdampfer sowie mit einem Rührreaktor zum Einsatz In weiteren Anwendungsbeispielen zum Thema Formaldehyd wird aufgezeigt dass sich die Online NMR Spektroskopie auch zum Studium komplexer Reaktionsnetzwerke zur Messung von Gaslöslichkeiten oder zur Quantifizierung kleinster Produktmengen unter schwierigen technischen Reaktionsbedingungen eignet Ferner werden Arbeiten zur Aufklärung und Quantifizierung der chemischen Prozesse bei der Absorption von Kohlendioxid in wässrigen Aminosäuren bei Drücken bis zu 30 bar und reaktionskinetische Untersuchungen von Veresterungen vorgestellt bei denen auch ein Vergleich mit einer GC Analytik durchgeführt wurde Ebenso wird gezeigt dass sich die Online NMR Spektroskopie zur Beobachtung von Reaktionen in ionischen Flüssigkeiten eignet Ein Teil der Arbeit beschäftigt sich mit der Untersuchung fluider Mischungen bei hohen Drücken Beispielfhaft werden Arbeiten zu Hochdruckgleichgewichten von Methanol in überkritischem Kohlendioxid vorgestellt die eine wertvolle experimentelle Datenbasis zur Überprüfung molekulardynamischer Modelle in der molekularen Simulation liefern Für die meisten Anwendungsbeispiele werden neue Reaktoren und Messapparaturen vorgestellt die sich besonders für den Einsatz in der Durchfluss NMR Spektroskopie eignen Schließlich wird kurz auf die Anwendung der NMR Spektroskopie zur Bestimmung physikalisch-chemischer Größen eingegangen wie z.B. zur Bestimmung von Diffusionskoeffizienten in technischen Mischungen Erstmals wird der Einsatz der Methode in der Taylor Dispersionstechnik beschrieben und experimentell belegt

Chemical Engineering and Chemical Process Technology - Volume I Ryszard Pohorecki, John Bridgwater, M. Molzahn, Rafiqul Gani and Crispulo Gallegos, 2010-11-30 Chemical Engineering and Chemical Process Technology is a theme component of Encyclopedia of Chemical Sciences Engineering and Technology Resources in the global Encyclopedia of Life Support Systems EOLSS which is an integrated compendium of twenty Encyclopedias Chemical engineering is a branch of engineering dealing with processes in which materials undergo changes in their physical or chemical state These changes may concern size energy content composition and or other application properties Chemical

engineering deals with many processes belonging to chemical industry or related industries petrochemical metallurgical food pharmaceutical fine chemicals coatings and colors renewable raw materials biotechnological etc and finds application in manufacturing of such products as acids alkalis salts fuels fertilizers crop protection agents ceramics glass paper colors dyestuffs plastics cosmetics vitamins and many others It also plays significant role in environmental protection biotechnology nanotechnology energy production and sustainable economical development The Theme on Chemical Engineering and Chemical Process Technology deals in five volumes and covers several topics such as Fundamentals of Chemical Engineering Unit Operations Fluids Unit Operations Solids Chemical Reaction Engineering Process Development Modeling Optimization and Control Process Management The Future of Chemical Engineering Chemical Engineering Education Main Products which are then expanded into multiple subtopics each as a chapter These five volumes are aimed at the following five major target audiences University and College students Educators Professional practitioners Research personnel and Policy analysts managers and decision makers and NGOs

Fundamentals of Water Pollution Daniel A. Vallero, 2024-09-27 Water Pollution Calculations Quantifying Pollutant Formation Transport Transformation Fate and Risks provides a comprehensive collection of relevant real world water pollution calculations The book's author explains in detail how to measure and assess risks to human populations and ecosystems exposed to water pollutants The text covers water pollution from a multivariate systems approach bringing in hydrogeological climatological meteorological processes health and ecological impacts and water and wastewater treatment and prevention After first reviewing the physics chemistry and biology of water pollution the author explores both groundwater and surface waters This is followed by an in depth look at water quality indicators measurements models and water engineering Groundwater remediation risk assessment and green engineering round out the text with forward thinking ideas towards sustainability This invaluable reference offers a practical tool for those needing a precise and applicable understanding of different types of water pollution calculations Includes applications of theory to real world problems with personalized and customized examples of calculations to prepare exams guidance documents and correspondence Walkthroughs and derivation of equations enhance knowledge so that complex water pollution concepts can be more easily grasped Explains processes and mechanisms providing an understanding of how pollutants are formed transported transformed deposited and stored in the environment

Air Pollution Calculations Daniel A. Vallero, 2023-09-17 Air Pollution Calculations Quantifying Pollutant Formation Transport Transformation Fate and Risks Second Edition enhances the systems science aspects of air pollution including transformation reactions in soil water sediment and biota that contribute to air pollution This second edition will be an update based on research and actions taken since 2019 that affect air pollution calculations including new control technologies emissions measurement and air quality modeling Recent court cases regulatory decisions and advances in technology are discussed and where necessary calculations have been revised to reflect these updates Sections discuss pollutant characterization pollutant transformation

and environmental partitioning Air partitioning physical transport of air pollutants air pollution biogeochemistry and thermal reactions are also thoroughly explored The author then carefully examines air pollution risk calculations control technologies and dispersion models The text wraps with discussions of economics and project management reliability and failure and air pollution decision making Provides real life current cases as examples of quantitation of emerging air pollution problems Includes straightforward derivation of equations giving practitioners and instructors a direct link between first principles of science and applications of technologies Presents example calculations that make scientific theory real for the student and practitioner

Chemical Production Scheduling Christos T. Maravelias, 2021-05-06 Understand common scheduling as well as other advanced operational problems with this valuable reference from a recognized leader in the field Beginning with basic principles and an overview of linear and mixed integer programming this unified treatment introduces the fundamental ideas underpinning most modeling approaches and will allow you to easily develop your own models With more than 150 figures the basic concepts and ideas behind the development of different approaches are clearly illustrated Addresses a wide range of problems arising in diverse industrial sectors from oil and gas to fine chemicals and from commodity chemicals to food manufacturing A perfect resource for engineering and computer science students researchers working in the area and industrial practitioners

Thermische Verfahrenstechnik Alfons Mersmann, Matthias Kind, Johann Stichlmair, 2006-03-30 Die Neuauflage h lt am bew hrten und anerkannten Konzept der Voraufgabe fest ein Grundlagenwerk zum Einsatz in der universit ren Lehre aber vor allem ein Nachschlagewerk f r den Beruf zum tieferen Verst ndnis der verfahrenstechnischen Zusammenh nge Gegen ber der Voraufgabe wurde das Werk allerdings erheblich erweitert sowie durch aktuelle Forschungsergebnisse und ein Kapitel zur konzeptuellen Prozessentwicklung erg nzt Die Grundverfahren werden auf der Basis der Thermodynamik der W rme und Stoff bertragung und der Fluidodynamik bergreifend dargestellt Damit erh lt der Praktiker erste Informationen ber die Auswahl die Auslegung und den Betrieb von Apparaten Nach einer komprimierten Darstellung wichtiger Grundlagen werden u a das Destillieren die Rektifikation die Absorption die Kristallisation und die Adsorption behandelt Es wird aufgezeigt wie sich grundlagenorientierte Berechnungsans tze f r fluiddynamische und kinetische Vorg nge in bekannten und neuen Apparaten finden lassen

Transport Phenomena Robert S. Brodkey, Harry C. Hershey, 2003-02 This book teaches the basic equations of transport phenomena in a unified manner and uses the analogy between heat transfer and mass and momentum to explain the more difficult concepts Part I covers the basic concepts in transport phenomena Part II covers applications in greater detail Part III deals with the transport properties The three transport phenomena heat mass and momentum transfer are treated in depth through simultaneous or parallel developments Transport properties such as viscosity thermal conductivity and mass diffusion coefficient are introduced in a simple manner early on and then applied throughout the rest of the book Advanced discussion is provided separately An entire chapter is devoted to the crucial material of non Newtonian phenomena This book covers heat transfer as it pertains to transport phenomena and covers mass

transfer as it relates to the analogy with heat and momentum The book includes a complete treatment of fluid mechanics for Ch E s The treatment begins with Newton s law and including laminar flow turbulent flow fluid statics boundary layers flow past immersed bodies and basic and advanced design in pipes heat exchanges and agitation vessels This text is the only one to cover modern agitation design and scale up thoroughly The chapter on turbulence covers not only traditional approaches but also includes the most contemporary concepts of the transition and of coherent structures in turbulence The book includes an extensive treatment of fluidization Computer programs and numerical methods are integrated throughout the text especially in the example problems

Thermal Separation Technology Alfons Mersmann,Matthias Kind,Johann Stichlmair,2011-07-28 Thermal Separation Technology is a key discipline for many industries and lays the engineering foundations for the sustainable and economic production of high quality materials This book provides fundamental knowledge on this field and may be used both in university teaching and in industrial research and development Furthermore it is intended to support professional engineers in their daily efforts to improve plant efficiency and reliability Previous German editions of this book have gained widespread recognition This first English edition will now make its content available to the international community of students and professionals In the first chapters of the book the fundamentals of thermodynamics heat and mass transfer and multiphase flow are addressed Further chapters examine in depth the different unit operations distillation and absorption extraction evaporation and condensation crystallization adsorption and chromatography and drying while the closing chapter provides valuable guidelines for a conceptual process development

Bioreaction Engineering Principles Jens Nielsen,John Villadsen,Gunnar Lidén,2012-12-06 This is the second edition of the text Bioreaction Engineering Principles by Jens Nielsen and John Villadsen originally published in 1994 by Plenum Press now part of Kluwer Time runs fast in Biotechnology and when Kluwer Plenum stopped reprinting the first edition and asked us to make a second revised edition we happily accepted A text on bioreactions written in the early 1990 s will not reflect the enormous development of experimental as well as theoretical aspects of cellular reactions during the past decade In the preface to the first edition we admitted to be newcomers in the field One of us JV has had 10 more years of job training in biotechnology and the younger author IN has now received international recognition for his work with the hottest topics of modern biotechnology Furthermore we are happy to have induced Gunnar Liden professor of chemical reaction engineering at our sister university in Lund Sweden to join us as co author of the second edition His contribution especially on the chemical engineering aspects of real bioreactors has been of the greatest value Chapter 8 of the present edition is largely unchanged from the first edition We wish to thank professor Martin Hjortso from LSU for his substantial help with this chapter

Industrial Chemical Separation Timothy C. Frank,Bruce S. Holden,2023-08-07 A fresh new treatment written by industry insiders this work gives readers a remarkably clear view into the world of chemical separation The authors review distillation extraction adsorption crystallization and the use of membranes providing historical perspective explaining key

features and offering insights from personal experience The book is for engineers and chemists with current or future responsibility for chemical separation on a commercial scale in its design operation or improvement or for anyone wanting to learn more about chemical separation from an industrial point of view The result is a compelling survey of popular technologies and the profession one that brings the art and craft of chemical separation to life Ever wonder how popular separation technologies came about how a particular process functions or how mass transfer units differ from theoretical stages Or perhaps you want some pointers on how to begin solving a separation problem You will find clear explanations and valuable insights into these and other aspects of industrial practice in this refreshing new survey **Transport**

Phenomena in Dispersed Media G. I. Kelbaliyev, D. B. Tagiyev, S.R. Rasulov, 2019-09-26 *Transport Phenomena in Dispersed Media* addresses the main problems associated with the transfer of heat mass and momentum The authors focus on the analytical solutions of the mass and heat transfer equations the theoretical problems of coalescence coagulation aggregation and fragmentation of dispersed particles the rheology of structured aggregate and kinetically stable disperse systems the precipitation of particles in a turbulent flow the evolution of the distribution function the stochastic counterpart of the mass transfer equations the dissipation of energy in disperse systems and many other problems that distinguish this book from existing publications **Key Selling Features** Covers all technological processes taking place in the oil and gas complex as well as in the petrochemical industry Presents new original solutions for calculating design as well as for the development and implementation of processes of chemical technology Organized to first provide an extensive review of each chapter topic solve specific problems and then review the solutions with the reader Contains complex mathematical expressions for practical calculations Compares results obtained on the basis of mathematical models with experimental data **Principles**

and Modern Applications of Mass Transfer Operations Jaime Benitez, 2016-12-08 A staple in any chemical engineering curriculum New edition has a stronger emphasis on membrane separations chromatography and other adsorptive processes ion exchange Discusses many developing topics in more depth in mass transfer operations especially in the biological engineering area Covers in more detail phase equilibrium since distillation calculations are completely dependent on this principle Integrates computational software and problems using Mathcad **Features** 25 30 problems per chapter *Food Process Design* Zacharias B. Maroulis, George D. Saravacos, 2003-05-09 Utilizes simplified computer strategies to analyze develop and optimize industrial food processes Discusses the integration and economic evaluation of the entire processing plant including effective use of water energy and raw materials process profitability and wastewater reduction Offers detailed numerical examples for major food processes including heating cooling evaporation dehydration and thermal processing *Physico-chemical Aspects of Textile Coloration* Stephen M. Burkinshaw, 2016-02-08 The production of textile materials comprises a very large and complex global industry that utilises a diverse range of fibre types and creates a variety of textile products As the great majority of such products are coloured predominantly using aqueous dyeing processes the

coloration of textiles is a large scale global business in which complex procedures are used to apply different types of dye to the various types of textile material The development of such dyeing processes is the result of substantial research activity undertaken over many decades into the physico chemical aspects of dye adsorption and the establishment of dyeing theory which seeks to describe the mechanism by which dyes interact with textile fibres Physico Chemical Aspects of Textile Coloration provides a comprehensive treatment of the physical chemistry involved in the dyeing of the major types of natural man made and synthetic fibres with the principal types of dye The book covers fundamental aspects of the physical and chemical structure of both fibres and dyes together with the structure and properties of water in relation to dyeing dyeing as an area of study as well as the terminology employed in dyeing technology and science contemporary views of intermolecular forces and the nature of the interactions that can occur between dyes and fibres at a molecular level fundamental principles involved in dyeing theory as represented by the thermodynamics and kinetics of dye sorption detailed accounts of the mechanism of dyeing that applies to cotton and other cellulosic fibres polyester polyamide wool polyacrylonitrile and silk fibres non aqueous dyeing as represented by the use of air organic solvents and supercritical CO₂ fluid as alternatives to water as application medium The up to date text is supported by a large number of tables figures and illustrations as well as footnotes and widespread use of references to published work The book is essential reading for students teachers researchers and professionals involved in textile coloration

Chromatographic Processes Roger-Marc Nicoud, 2015-04-20 A result oriented practical guide to key approaches methodologies and tools for designing modelling and simulating chromatographic processes

Mixing Process Technology Kishore Kar, Richard Cope, Juergen Lueske, 2025-07-31 Industrial mixing processes often present multiple optimization challenges to producing desirable products The resulting processes must be cost effective first time right and frequently the designated most effective technology for the global manufacture of specific products *Mixing Process Technology A Guide to Industrial Applications* shares the authors extensive knowledge of mixing research and industrial practice It features 20 industrial mixing chapters that are purposely light on mixing fundamentals while heavy on practical mixing applications for practical process design and manufacturing This text serves as an applied guide to industrial mixing for practitioners who want brief explanations of mixing concepts with real life examples and software to help perform associated design calculations This book also Offers side by side discussion of mixing systems including impellers and rotor stators as offered by several major manufacturers Describes the authors innovative mixer designs to meet manufacturing needs Includes a chapter by a mixer manufacturing representative describing design sizing and expensing of industrial mixers Presents a chapter by a mixing equipment manufacturing leader that explains mechanical design considerations in clear terms Contains a chapter on emerging mixing technologies including mixing via resonant acoustics and controlled cavitation Discusses computational fluid dynamics in mixing with multiple practical examples by a contributing author from a leading pharmaceutical company Includes Excel based mixing worksheets

throughout book examples and Excel based input output mixit io interface hosted on the publisher s website This book is aimed at chemical and process engineers as well as students seeking to understand industrial mixing technology

Information Sources in Engineering Roderick A. Macleod,Jim Corlett,2012-04-17 The current thoroughly revised and updated edition of this approved title evaluates information sources in the field of technology It provides the reader not only with information of primary and secondary sources but also analyses the details of information from all the important technical fields including environmental technology biotechnology aviation and defence nanotechnology industrial design material science security and health care in the workplace as well as aspects of the fields of chemistry electro technology and mechanical engineering The sources of information presented also contain publications available in printed and electronic form such as books journals electronic magazines technical reports dissertations scientific reports articles from conferences meetings and symposiums patents and patent information technical standards products electronic full text services abstract and indexing services bibliographies reviews internet sources reference works and publications of professional associations *Information Sources in Engineering* is aimed at librarians and information scientists in technical fields as well as non professional information specialists who have to provide information about technical issues Furthermore this title is of great value to students and people with technical professions

Absorption-Based Post-Combustion Capture of Carbon Dioxide Paul Feron,2016-05-27 Absorption Based Post Combustion Capture of Carbon Dioxide provides a comprehensive and authoritative review of the use of absorbents for post combustion capture of carbon dioxide As fossil fuel based power generation technologies are likely to remain key in the future at least in the short and medium term carbon capture and storage will be a critical greenhouse gas reduction technique Post combustion capture involves the removal of carbon dioxide from flue gases after fuel combustion meaning that carbon dioxide can then be compressed and cooled to form a safely transportable liquid that can be stored underground Provides researchers in academia and industry with an authoritative overview of the amine based methods for carbon dioxide capture from flue gases and related processes Editors and contributors are well known experts in the field Presents the first book on this specific topic

Continuous Biopharmaceutical Processes David Pfister,Lucrèce Nicoud,Massimo Morbidelli,2018-10-11 Provides a coherent and critical view on the potential benefits of various continuous processes in the biopharmaceutical industry

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