Differential Scanning Calorimetry As A Tool For Analysis

As a result of the Process Analytical Technologies (PAT) initiative launched by the U.S. Food and Drug Administration (FDA), analytical development is receiving more attention within the pharmaceutical industry. Illustrating the importance of analytical methodologies, Thermal Analysis of Pharmaceuticals presents reliable and versatile charac

Here, researchers review the latest breakthroughs in protein research. Their contributions explore emerging principles and techniques and survey important classes of proteins that will play key roles in the field's future. Articles examine the possibility of a Boltzman-like distribution in protein substructures, the new technique of Raman spectroscopy, and compact intermediate states of protein folding. This well-illustrated volume also features coverage of proteins that bind nucleic acids.

The use of thermal and calorimetric methods has shown rapid growth over the past few decades, in an increasingly wide range of applications. The original text was published in 2001; since then there have been significant advances in various analytical techniques and their applications. This second edition supplies an up to date, concise and readable account of the principles, experimental apparatus and practical procedures used in thermal analysis and calorimetric methods of analysis. Written by experts in their field, brief accounts of the basic theory are reinforced with detailed technical advances and contemporary developments. Where appropriate, applications are used to highlight particular operating principles or methods of interpretation. As an important source of information for many levels of readership in a variety of areas, this book will be an aid for students and lecturers through to industrial and laboratory staff and consultants.

Calorimetry, as a technique for thermal analysis, has a wide range of applications which are not only limited to studying the thermal characterisation (e.g. melting temperature, denaturation temperature and enthalpy change) of small and large drug molecules, but are also extended to characterisation of fuel, metals and oils. Differential Scanning Calorimetry is used to study the thermal behaviours of drug molecules and excipients by measuring the differential heat flow needed to maintain the temperature difference between the sample and reference cells equal to zero upon heating at a controlled programmed rate. Microcalorimetry is used to study the thermal transition and folding of biological macromolecules in dilute solutions. Microcalorimetry is applied in formulation and stabilisation of therapeutic proteins. This book presents research from all over the world on the applications of calorimetry on both solid and liquid states of materials.

The wide range of applications of thermal methods of analysis in measuring physical properties, studying chemical reactions and determining the thermal behaviour of samples is of interest to academics and to industry. These applications prompted the writing of this book, in the hope that the descriptions, explanations and examples given would

be of help to the analyst and would stimulate the investigation of other thermal techniques. Thermal studies are a fascinating means of examining the samples and the problems brought to us by colleagues, students and clients. If time allows, watching crystals change on a hot-stage microscope, or measuring the properties and changes on a DSC or TG or any thermal instrument can be a rewarding activity, besides providing valuable analytical information. This book started from a series of lectures delivered at Kingston University and at meetings of the Thermal Methods Group of the United Kingdom. The collaboration and information supplied to all the contribu tors by colleagues and instrument manufacturers is most gratefully ack nowledged, as are the valuable contributions made at meetings of the International Confederation for Thermal Analysis and Calorimetry (ICT AC) and at the European Symposia on Thermal Analysis and Calorimetry (ESTAC).

Plastics, Heat measurement, Scanners, Thermal analysis, Differential thermal analysis, Quantitative analysis, Thermal measurement, Specific heat, Plastics and rubber technology

The authors show how DSC can be applied to various fields of polymers science where other methods have been unsuccessful. They discuss the ways in which DSC facilitates quantitative studies of the thermodynamic parameters and kinetics of melting, crystallization, liquid-crystallization, and different phase and relaxation transitions.

Differential Scanning Calorimetry (DSC) is a well established measuring method which is used on a large scale in different areas of research, development, and quality inspection and testing. Over a large temperature range, thermal effects can be quickly identified and the relevant temperature and the characteristic caloric values determined using substance quantities in the mg range. Measurement values obtained by DSC allow heat capacity, heat of transition, kinetic data, purity and glass transition to be determined. DSC curves serve to identify substances, to set up phase diagrams and to determine degrees of crystallinity. This book provides, for the first time, an overall description of the most impor tant applications of Differential Scanning Calorimetry. Prerequisites for reliable measurement results, optimum evaluation of the measurement curves and esti mation of the uncertainties of measurement are, however, the knowledge of the theoretical bases of DSC, a precise calibration of the calorimeter and the correct analysis of the measurement curve. The largest part of this book deals with these basic aspects: The theory of DSC is discussed for both heat flux and power compensated instruments; temperature calibration and caloric calibration are described on the basis of thermodynamic principles. Desmearing of the measurement curve in different ways is presented as a method for evaluating the curves of fast transitions.

MTDSC provides a step-change increase in the power of calorimetry to characterize virtually all polymer systems including curing systems, blends and semicrystalline polymers. It enables hidden transitions to be revealed, miscibility to be accurately assessed, and phases and interfaces in complex blends to be quantified. It also enables crystallinity in complex systems to be measured and provides new insights into melting behaviour. All of this is achieved by a simple modification of conventional DSC. In 1992 a new calorimetric technique was introduced that superimposed a small modulation on top of the conventional linear temperature program typically used in differential scanning calorimetry. This was combined with a method of data analysis that enabled the sample’s response to the linear component of the temperature program to be separated from its response to the periodic component. In this way, for the first time, a signal equivalent to that of

conventional DSC was obtained simultaneously with a measure of the sample's heat capacity from the modulation. The new information this provided sparked a revolution in scanning calorimetry by enabling new insights to be gained into almost all aspects of polymer characteristics. This book provides both a basic and advanced treatment of the theory of the technique followed by a detailed exposition of its application to reacting systems, blends and semicrystalline polymers by the leaders in all of these fields. It is an essential text for anybody interested in calorimetry or polymer characterization, especially if they have found that conventional DSC cannot help them with their problems.

Thermal Analysis techniques are used in a wide range of disciplines, from pharmacy and foods to polymer science, materials and glasses; in fact any field where changes in sample behaviour are observed under controlled heating or controlled cooling conditions. The wide range of measurements possible provide fundamental information on the material properties of the system under test, so thermal analysis has found increasing use both in basic characterisation of materials and in a wide range of applications in research, development and quality control in industry and academia. Principles and Applications of Thermal Analysis is written by manufacturers and experienced users of thermal techniques. It provides the reader with sound practical instruction on how to use the techniques and gives an up to date account of the principle industrial applications. By covering basic thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) including the new approach of Fast Scanning DSC, together with dynamic mechanical analysis (DMA /TMA) methods, then developing the discussion to encompass industrial applications, the book serves as an ideal introduction to the technology for new users. With a strong focus on practical issues and relating the measurements to the physical behaviour of the materials under test, the book will also serve as an important reference for experienced analysts.

In this chapter we briefly introduce the main physical principles of DSC as well as related techniques. After a quick survey of the more common experimental techniques, we describe the thermodynamics and kinetics of events accompanying a heating/cooling process. We focus on lipid membranes of one or more components. Both the thermotropic and the barotropic behaviours are investigated, as well as the water/lipid ratio. The effect of foreign impurities (hydrophobic molecules, proteins) dissolved in the lipid matrix on DSC thermotropic behaviour is also investigated, either in the ideal mixing model or for non-ideal miscibility. In the poor miscibility limit, lipids and hydrophobic impurities may undergo phase separation. The mechanisms of phase separation are discussed and related to experimental DSC features. Out-of-equilibrium phenomena, such as the different thermotropic behaviour between heating and cooling modes or the kinetics of lipid/water partitioning, are explained using simple models for phase transitions.

Plastics, Heat measurement, Scanners, Thermal analysis, Differential thermal analysis, Oxidation methods

This book resolves the need for a practise-oriented guide to differential scanning calorimetry (DSC), the most frequently used technique in thermal analysis. The authors describe almost all types of the respective instrumentation on the market and provide the newcomer and the experienced practitioner with a comprehensive insight into theory and application of DSC methods.

With a focus on structure-property relationships, this book describes how polymer morphology affects properties and how scientists can modify them. The book covers structure development, theory, simulation, and processing; and discusses a broad range of techniques and methods. • Provides an up-to-date, comprehensive introduction to the principles and practices of polymer

morphology • Illustrates major structure types, such as semicrystalline morphology, surface-induced polymer crystallization, phase separation, self-assembly, deformation, and surface topography • Covers a variety of polymers, such as homopolymers, block copolymers, polymer thin films, polymer blends, and polymer nanocomposites • Discusses a broad range of advanced and novel techniques and methods, like x-ray diffraction, thermal analysis, and electron microscopy and their applications in the morphology of polymer materials

In the past decades, the scan rate range of calorimeters has been extended tremendously at the high end, from approximately 10 up to 10 000 000 °C/s and more. The combination of various calorimeters and the newly-developed Fast Scanning Calorimeters (FSC) now span 11 orders of magnitude, by which many processes can be mimicked according to the time scale(s) of chemical and physical transitions occurring during cooling, heating and isothermal stays in case heat is exchanged. This not only opens new areas of research on polymers, metals, pharmaceuticals and all kinds of substances with respect to glass transition, crystallization and melting phenomena, it also enables in-depth study of metastability and reorganization of samples on an 1 to 1000 ng scale. In addition, FSC will become a crucial tool for understanding and optimization of processing methods at high speeds like injection molding. The book resembles the state-of-the art in Thermal Analysis & Calorimetry and is an excellent starting point for both experts and newcomers in the field.

Discover a comprehensive exploration of recent progress in the preparation of nitroalkanes from two leading voices in the field Nitroalkanes: Synthesis, Reactivity, and Applications delivers a thorough summary of the importance of nitroalkanes in organic synthesis. The book covers their preparation, transformation into other functional groups, like carbonyls and amines, and their use in the formation of single carbon-carbon or double carbon-carbon bonds. The distinguished authors have included chapters on acyclic and cyclic alpha-nitro ketones as well as the synthesis of cyclopropanes and spiro ketals. The book provides treatments of the application of nitroalkanes for the synthesis of important heterocycles, poly-functionalized structures, natural products, and compounds of biological and pharmaceutical interest. A one-stop resource in a topic that hasn???t been fully addressed by any other book in decades, this book covers the most important synthetic routes toward nitroalkanes. Readers will also benefit from the inclusion of: A thorough introduction to the synthesis of nitroalkanes, as well as the transformation of the nitro group into other functionalities An exploration of the formation of C-C single bonds, C=C double bonds, and the breaking of C3C bonds from cyclic alpha-nitro ketones Discussions of acyclic alpha-nitro ketones, nitroalkanes as precursors of cyclopropanes, and the synthesis of spiro ketals An examination of the preparation and synthetic applications of 1,3-Dinitroalkanes Perfect for organic chemists, natural products chemists, and catalytic chemists, Nitroalkanes: Synthesis, Reactivity, and Applications will also earn a place in the libraries of medicinal chemists seeking a one-stop resource for the most recent developments in the preparation of nitroalkanes, their functionalization, and their applications.

Plastics, Heat measurement, Scanners, Thermal analysis, Differential thermal analysis, Crystallization, Kinetics, Thermoplastic polymers, Polymers

Differential Scanning Calorimetry: Applications in Fat and Oil Technology provides a complete summary of the scientific literature about differential scanning calorimetry (DSC), a well-known thermo-analytical technique that currently has a large set of applications covering several aspects of lipid technology. The book is divided into three major sections. The first section covers the applications of DSC to study cooling and heating profiles of the main source of oils and fats. The second is more theoretical, discussing the application of DSC coupled to related thermal techniques and other physical measurements. And the third covers specific applications of DSC in the field of quality evaluation of palm, palm kernel, and coconut oils and their fractions as well as of some other important aspects of lipid technology such as shortening and margarine functionality, chocolate technology, and food emulsion stability. This book is a helpful resource for academicians, food scientists, food engineers and technologists, food industry operators, government researchers, and regulatory agencies.

In this updated and fully revised second edition, the authors provide the newcomer and the experienced practitioner with a balanced and comprehensive insight into all important methods and aspects of Differential Scanning Calorimetry (DSC), including a sound presentation of the theoretical basis of DSC thermal analysis and temperature-modulated DSC (TMDCS). Emphasis is placed on modern evaluation techniques, instrumentation, the underlying measurement principles, metrologically correct calibrations, factors influencing the measurement process, and on the exact interpretation of the results. The information enables the research scientist, the analyst and experienced laboratory staff to choose the most suitable equipment, to apply DSC methods successfully, to interpret the measurement curve, and thus to measure key properties precisely. In addition, the new edition includes improved instrumental techniques such as Tzerotm and StepScantm, new evaluation techniques, more applications, and the latest references. Calorimetry - Dynamische Differenzkalorimetrie - Thermal Analysis - Thermische Analyse

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Solvates -- References -- 4 Pharmaceutical Salts -- 4.1 Introduction -- 4.2 Importance of Pharmaceutical Salts -- 4.3 Weak Acid, Weak Base, and Salt -- 4.4 pH-Solubility Profiles of Ionizable Compounds

Containing the very latest information on all aspects of enthalpy and internal energy as related to fluids, this book brings all the information into one authoritative survey in this well-defined field of chemical thermodynamics. Written by acknowledged experts in their respective fields, each of the 26 chapters covers theory, experimental methods and techniques and results for all types of liquids and vapours. These properties are important in all branches of pure and applied thermodynamics and this vital source is an important contribution to the subject hopefully also providing key pointers for cross-fertilization between sub-areas.

In this fully updated and revised second edition the authors provide the newcomer and the experienced practitioner with a balanced and comprehensive insight into all important DSC methods, including a sound presentation of the theoretical basis of DSC and TMDSC measurements. Emphasis is layed on instrumentation, the underlying measurement principles, metrologically correct calibrations, factors influencing the measurement process, and on the exact interpretation of the results. The information given enables the research scientist, the analyst and experienced laboratory staff to apply DSC methods successfully and to measure respective properties correctly.

Presents a solid introduction to thermal analysis, methods,instrumentation, calibration, and application along with thenecessary theoretical background. Useful to chemists, physicists, materials scientists, andengineers who are new to thermal analysis techniques, and toexisting users of thermal analysis who wish expand their experienceto new techniques and applications Topics covered include Differential Scanning Calorimetry andDifferential Thermal Analysis (DSC/DTA), Thermogravimetry,Thermomechanical Analysis and Dilatometry, Dynamic MechanicalAnalysis, Micro-Thermal Analysis, Hot Stage Microscopy, andInstrumentation. Written by experts in the various areas of thermalanalysis Relevant and detailed experiments and examples follow eachchapter.

Polyether and polyester urethane elastomers were aged in humidity (98%), water, and air at 160F, 170F, and 185F for 7, 14, 28, 42, 70, and 100 days. After each aging condition, tensile strength of the materials was determined and Differential Scanning Calorimetry (DSC) traces were run to observe changes in thermal transitions of the aged specimens. An effort was made to relate these transitions with actual reduction in tensile strength. Results were evaluated using DSC as a method for indicating hydrolytic degradation of the urethane elastomers studied in this investigation. Keywords: Thermal analysis; Endothermic; Glass transition temperature (Tg); Hydrolysis; Paracrystalline domain. (aw).