

Developments in polymer characterization

Edited by J. V. Dawkins

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During the last decade, an increasing number of high-quality advanced texts, dealing with various aspects of polymer science, have been published. It is probable that the present multi-author volume, which is the first of a series, will come to be regarded as among the best of these books. The editor, John Dawkins, is one of the most dynamic of the younger generation of British polymer chemists. As a result the volume is organized and edited with a confidence bred by day to day confrontation with the real problems of polymer characterization. This does much to minimize for the reader the obvious problems of variation in style and presentation which occur when several authors collaborate.

Multiple authorship is today essential in any authoritative work on polymer characterization because the methods available are now so diverse. Classically, characterization involved exclusively the investigation of solution properties in order to determine molecular weights and molecular volumes. Later studies on stereoregular polymers stimulated the development of methods for characterizing chain structure. The discovery of polymer crystals and the development of multiphase polymeric systems, for example gels, blends and composites, have led to the inception of powerful new techniques applicable to bulk properties. The description of these techniques by specialists, several of whom are acknowledged internationally as among the leaders in their fields, ensures the uniform high standard of this book. Moreover, it leaves it to the individual reader to assess the relative value and applicability of the methods to their own particular problems.

Two chapters, one on ^{13}C n.m.r. spectroscopy and the other on small angle neutron scattering, deal with new methods which have been developed mainly during the past five years. Both accounts include a useful introductory discussion of the theoretical principles involved. The chapter on small

angle neutron scattering, by R. W. Richards, is topical because the method has been applied recently to a number of fundamental problems in polymer science. Thus, results on bulk polymers have led to direct proof of unperturbed dimensions in the amorphous state and facilitated determination of polymer dimensions in concentrated solution. This has done much to further developments in polymer solution theory.

The lucid chapter on ^{13}C n.m.r. by A. V. Cunliffe, reviews the detailed application of this technique to structural studies at high resolution and to polymer mobility investigations *via* relaxation phenomena. Also examined are the exciting prospects of ^{13}C n.m.r. studies in the solid state. With full proton decoupling, the low abundance of ^{13}C , ensures that carbon peaks appear as single lines without the complication of spin-spin interaction between adjacent nuclei. This, together with the extended chemical shift range of ^{13}C n.m.r. as compared to proton n.m.r., ensures that the resolution of carbon atoms in different molecular environments greatly exceeds that of protons. The detailed structural information on polymers which may be obtained from ^{13}C n.m.r. studies is correspondingly much greater. For example, useful ^{13}C n.m.r. spectra may be obtained of solid polymers above the glass transition temperature, a condition which applies for solid elastomers, and of swollen gels of rigid polymers. With the advent of dedicated computers of low cost, the price of Fourier transform n.m.r. spectrometers is falling. Soon, ^{13}C n.m.r. spectrometers will fall within the budget of most polymer laboratories. This excellent chapter will do much to popularize the method.

Despite the importance of mass spectrometry in synthetic organic chemistry, this technique is not widely employed in polymer characterization. A useful chapter, by R. D. Sedgwick, familiarizes the reader with electron impact methods of studying oligomers obtained by polymer degradation. The examination of both externally pyrolysed and direct, *in situ*, pyrolysed materials are discussed. Briefly reviewed are secondary ion mass spectrometry, laser pyrolysis mass spectrometry and macromass spectrometry. The latter, pioneered by M. Dole, has the

potential to obtain molecular weight distributions directly and depends on obtaining isolated charged macromolecules in the vapour state by means of an electrospray system.

A major part of the book is devoted to important recent developments which have taken place in established characterization methods. J. V. Dawkins reviews high performance gel permeation chromatography with emphasis on the use of microparticulate chromatography packings to facilitate high speed determination of molecular weight distribution and average molecular weights. An account of the use of light scattering, by A. J. Hyde, concentrates mainly on Rayleigh line broadening measurements for diffusion coefficients of polymers in solution, Brillouin scattering for transition temperatures on solids, and scattering measurements in the region between the binodal and spinodal in polymer solutions. These are areas where there have been significant developments in recent years. Characterization of two phase polymeric systems by small-angle X-ray scattering is discussed by D. S. Brown and R. E. Wetton. A useful chapter by M. J. Richardson, on differential scanning calorimetry, deals with quantitative thermodynamic interpretation of the glass transition, crystallinity and heat of fusion.

A final chapter, by D. Hemsley, collects together much information on characterization of polymer surfaces by both optical and electron microscopy. Modern and traditional methods of sample preparation are discussed with emphasis placed on the results obtainable by maximising image contrast. A number of useful illustrative examples of current applications are included.

To summarize, this book will appeal widely as a concise reference work of lasting usefulness to all those with a commitment to polymer science and polymer technology. Much important information is included which is not readily abstracted from the original scientific journals. The book is recommended warmly as the best account now available of recent advances in polymer characterization. We look forward to the publication of further volumes in this series.

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