

Size Exclusion Chromatography

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Size exclusion chromatography (SEC), also referred to as gel permeation, gel filtration, steric exclusion, or gel chromatography, is an entropically controlled separation technique that depends on the relative size or hydrodynamic volume of a macromolecule with respect to the average pore size of the packing. With a properly calibrated SEC column, all the statistical average molecular weights of a polymer can be determined, as well as the molecular weight distribution. Absolute molecular weight measurements are possible with the use of either an on-line light-scattering detector without column calibration or an on-line viscometer with universal calibration. With these detectors, it is possible also to determine molecular conformation and long-chain branching. In addition to obtaining molecular parameters, SEC is useful also for preparative fractionation of polymers and for separating small molecules from complex polymeric or biogenic matrixes as an aid to sample cleanup.

SEC has become a mature and well-accepted technique for characterizing both synthetic polymers and biopolymers. Advances in SEC packings include the use of mixed-bed columns, allowing a wide molecular weight separation range with fairly linear calibration. For protein separations, composite packings, such as the Superdex series, are increasing in popularity because of their high pore volume, inertness, and small particle size availability. There continues to be less emphasis placed on theoretical aspects of SEC, including band broadening, and more focus on applications, especially the use of on-line light-scattering detectors and viscometers. With these molecular weight-sensitive detectors, one can now determine branching, molecular size, and conformation as a function of molecular weight in a single analysis. The use of both of these detectors, together with a concentration-sensitive detector, has greatly improved the accuracy and precision of these measurements. More protein laboratories are now combining Stokes radius measurements, obtained by SEC, with sedimentation coefficient data from ultracentrifugation to study protein hydrodynamics, especially protein folding properties.

We see increasing numbers of publications on the determination of compositional heterogeneity of oligomers and polymers using SEC with on-line spectrophotometric detection, such as UV. The most important advancement in this area is the use of mass spectrometry for characterizing both the absolute molecular weight and the chemical composition of polymers and biopolymers. Although not covered extensively in this review, the use of matrix-assisted laser desorption/ionization mass spectrometry

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is being used off-line for the molecular weight characterization of oligomers and biopolymers.

The use of interactive HPLC (adsorption, precipitation, and critical condition chromatography) for separating polymers and oligomers on the basis of chemical composition has continued to increase. Supercritical fluid chromatography and extraction, temperature-rising elution fractionation, and thermal and flow field-flow fractionation techniques are beginning to play a major role in polymer separations. Although these methods are non-SEC separations, they are included in this review because of their importance.

In terms of applications, SEC has proven valuable for the characterization of block copolymer micelles, as well as for the determination of biopolymer association, ligand binding, and conformational changes. Long-chain branching measurement as a function of molecular weight is now a routine determination with on-line viscometry. With the proper choice of mobile phase and packing type, most polymers and biopolymers can be characterized by SEC on a routine basis. In many laboratories, SEC has now supplanted osmometry and off-line light-scattering measurements for the determination of number- and weight-average molecular weights.

This review covers fundamental developments in SEC (and HPLC of polymers) abstracted by *Chemical Abstracts* and *Medline* from 1994 and 1995 inclusive and is a continuation of our previous review (*B1*). Topics in this review are listed in the Table of Contents. Suggestions are always welcome for improving our coverage of SEC and related techniques. Also, please contact the corresponding author (H.G.B.) if we have missed any significant study, which we will include in our next review.

Writing these reviews is a long and laborious process that involves developing search routines, reading all abstracts and often full papers, and categorizing this information into a convenient format. To this end, we are grateful to Mr. Douglas A. Eckel for improving and carrying out our literature search strategy. The manuscript was typed with skill and patience by Mrs. Rebecca Pennington, to whom we are thankful. Thanks also goes to Mr. Bud Permar for his countless trips to the library. We also thank our respective companies for their support and encouragement in the preparation of the manuscript.

BOOKS AND SYMPOSIA

Wu (*A1*) edited *Handbook of Size Exclusion Chromatography*, consisting of contributed chapters dealing with SEC packings, detectors, and analysis of specific classes of polymers, such as polyamides, polyesters, elastomers, asphalts, poly(vinyl alcohol), polyacrylamide, poly(vinyl acetate), cellulose, starch, lignin, proteins, and nucleic acids. Contributions from a symposium, Hyphenated Techniques in Polymer Characterization, held at the 206th National Meeting of the American Chemical Society, Chicago, IL, August 22–27, 1993, appeared in two ACS publications (*A2*, *A3*).

A new journal, *International Journal of Polymer Analysis and Characterization*, which is devoted to the analytical aspects of polymer science, launched its first issue in 1995 (*A4*). Proceedings of the Sixth and Seventh International Symposia on Polymer Analysis and Characterization (ISPAC) have been published in volumes 1 and 2 of this journal (*A5*, *A6*). The proceedings of ISPAC-8, held in Sanibel Island, FL, May 22–24, 1995, are in press.

Contributions from the 1994 International GPC Symposium, held June 5–8, 1994, in Lake Buena Vista, FL, have been issued (*A7*).

GENERAL REVIEWS

Reviews of specific topics are covered in the appropriate sections of this article. The Selected Applications section lists specific reviews based on polymer type. Barth and colleagues (*B1*) presented a comprehensive coverage of the SEC literature from 1992 to 1993; the present review is a continuation of that format. Malawer (*B2*) wrote an introductory chapter on the fundamentals of SEC, including calibration approaches.

General reviews were written on the principles and applications of SEC (*B3–B5*), including articles in French (*B6*), Japanese (*B7–B9*), and Korean (*B10*, *B11*). Barth (*B12*) presented an overview of recent developments in the use of hyphenated multidimensional separation techniques for the characterization of polymers, with emphasis on molecular weight-sensitive detectors. Urban (*B13*) discussed synergistic aspects of hyphenated methods for polymer analysis.

THEORY

Degoulet et al. (*C1*) used Monte Carlo methods to evaluate the effect of pore shape and polymer chain flexibility on the steric partition coefficient, K . They defined the chromatographic radius, R_c , of the macromolecule as the radius of a sphere with the same value of K . They found that, for a given pore geometry, the ratio of R_c to the viscosity hydrodynamic radius, R_η , was not strictly independent of the flexibility and the relative thickness of the macromolecule. Deviations from universal calibration are expected when the global flexibility of the sample polymer is different from that of the calibration standard, or when the polymer contour length is small relative to the persistence length. McCoy (*C2*) presented a mathematical theory for the separation of distributions of solutes in cylindrical pores with discrete and continuous size distributions.

Harlan et al. (*C3*) described a method for calibration of columns based on the assumption that the pore size distribution can be described by two Gaussian distributions, which accounts for the nonlinear relationship between the Stokes radius, R_s , and the partition coefficient. They successfully applied the method to low-pressure chromatographic gels. Kuntz et al. (*C4*) evaluated models of the SEC separation of asymmetric solutes using rigid rodlike and spherical solutes (schizophyllan and Ficoll). They found that neither the equilibrium "random pore" model nor the "end-on insertion" model was compatible with the results, and they hypothesized that the dynamics of solute motion limit equilibrium models.

The resolving power and separation time in thermal field-flow fractionation (TFFF), packed column and open-tubular hydrodynamic chromatography, and SEC were compared by Stegeman et al. (*C5*). TFFF performed best for the separation of high-molecular-weight compounds, and SEC was considered the most suitable method for molecular weights below 100 000.

Mrkvicková et al. (*C6*) compared size distribution data from dynamic light scattering with that obtained from SEC. Adrover et al. (*C7*) analyzed the dispersion properties of biomolecules in SEC both theoretically and experimentally, and Martys (*C8*) used computational methods to numerically simulate hydrodynamic dispersion in random porous media and developed a simple model of SEC.

BAND BROADENING

Lederer et al. (*D1*) studied band broadening in SEC of narrow-molecular-weight-distribution polystyrenes and polypropylene and found that the elution volume dependence of the peak broadening was different for the two polymers. They concluded that band broadening parameters should be determined with narrow molecular weight distributions of samples of the polymer to be analyzed. A deconvolution technique, separating sample polydispersity from peak broadening and improving the resolution of SEC analysis of lipoproteins, was presented by Barbee et al. (*D2*). Chen et al. (*D3*) described a method for correcting nonlinear calibration curves for band broadening.

CALIBRATION

General. Acevedo et al. (*E1*) showed how the discotic shape of asphaltene could be derived from the differences in SEC calibration curves between polystyrene and octylated asphaltene standards. Nali and Manclossi (*E2*) also characterized asphaltene molecular weight distributions using SEC and vapor pressure osmometry. By combining the results, they were also able to study intermolecular associations. Martin and Balke (*E3*) compared the effect of the cyclic oligomer peak in the molecular weight distribution of poly(ethylene terephthalate) on different calibration methods.

Bignotti et al. (*E4*) used NMR to determine the number-average molecular weight of a series of poly(amido amines), which were then used as SEC calibration standard in a study of their degradation behavior. Komatsu et al. (*E5*) and Ahsan et al. (*E6*, *E7*) studied different calibration standards and methods for characterizing low molecular weight heparins. Barnikol and Poetschke (*E8*) showed both that human thrombocytes could be used as a universal indicator of the column interstitial volume in SEC and also that Vineyard snail haemocyanin is a useful high-molecular-weight globular protein calibrant.

Krol and Dejnega (*E9*) used model urethane oligomer standards as calibrants for polyurethane molecular weight analysis, and Boutevin et al. (*E10*) used acrylic acid telomers as calibration standards for analysis of acrylic acid and thioglycolic acid. Poly(ethylene oxide) was characterized using oligo(ethylene glycols) as standards by Kinugasa et al. (*E11*). Calibration procedures for coal liquids (*E12*) and for humic substances (*E13*) were also presented.

Wu et al. combined off-line dynamic laser light-scattering measurements with SEC to establish a hydrodynamic volume universal calibration curve (*E14*) in order to characterize hydroxyethyl cellulose acetate and to study chain conformation (*E15*).

Universal Calibration. Giddings (*E16*) discussed and contrasted universal calibration in SEC and TFFF. In SEC, universal calibration makes it possible to transfer calibration data obtained using polymer standards to the analysis of other polymers of interest. The calibration curve is column dependent. In TFFF, universal calibration is based on physicochemical constants and, in principle, is system independent. Tsitsilianis and Ktoridis (*E17*) described a method for characterizing the number of arms in star polymers using universal calibration and only a single concentration detector. Dubin (*E18*) showed that nonionic polysaccharides are ideal calibration standards for aqueous SEC; in addition, they provide a useful method for studying the relationship between the partition coefficient in SEC and macromolecular size.

Nave et al. (*E19*) used universal calibration to characterize high molecular weight proteins. They extended the range of their calibration standards by using both a denaturing aqueous phase and a buffered solution. They found that, on certain columns, the universal calibration curve was congruent for native and denaturing conditions. Mourey and Balke (*E20*) proposed a simple method for using SEC with both concentration and light-scattering detectors to calibrate other SEC systems having only concentration detectors. The method was demonstrated for poly(ethylene terephthalate). The light-scattering detector was used to establish a correlation between the molecular weight of polystyrene and poly(ethylene terephthalate) at each elution volume, and this correlation was employed on other single-detector systems.

Universal calibration was also used to characterize ultrahigh-molecular-weight polyethylene (*E21*), butyl rubbers (*E22*) and acrylonitrile-butadiene rubber (*E23*), poly(propylene carbonate) and poly(ethylene carbonate) (*E24*), Chinese lacquer polysaccharide (*E25*, *E26*), and chitosan (*E27*).

Data Handling. Nwankwo and Abbott (*E28*) presented a simplified method for linear calibration using broad-molecular-weight-distribution standards. Duggleby (*E29*) described a method for calculating the best calibration curve, linear or curved, for data from a series of standards and for estimating the accuracy of the curve at a given molecular weight value. Atiquallah et al. (*E30*) described an algorithm for optimizing the calculation of the molecular weight distribution of poly(vinyl chloride) using a polystyrene calibration curve and membrane osmometry to measure the number-average molecular weight. Shortt (*E31*) reviewed methods of calculating the differential molecular weight distribution in SEC, and Crow (*E32*) discussed using calibration data as a diagnostic tool for trouble-shooting an SEC system.

Rosset et al. (*E33*) discussed calibration methods and the experimental conditions that must be fulfilled in order to determine accurate molecular weight distributions. Regehr et al. (*E34*) published a paper illustrating the use of ensemble averaging and digital filtering in liquid-phase separation techniques including SEC. Kilz described a new software program for SEC analysis (*E35*).

The Japanese Society for Analytical Chemistry published the results of a round robin test on molecular weight distribution determination by SEC (*E36*). They found that the relative standard deviations in the molecular weight averages at each laboratory were between 1 and 3%. However, among the 26 laboratories, the relative standard deviations were 16–18% for the number-average molecular weight and 7–10% for the weight-average molecular weight.

NON-SIZE-EXCLUSION EFFECTS

Shear Degradation. Shalliker et al. (*F1*) investigated the use of large-diameter C18 pellicular particles for reversed-phase HPLC of ultrahigh molecular weight ($>7 \times 10^6$) polystyrenes and claimed no noticeable degradation. Gur'yanova et al. (*F2*) discussed problems associated with SEC of ultrahigh-molecular-weight ($>2 \times 10^6$ – 3×10^6) polymers. Using a low-angle light-scattering detector, they found that the SEC of a 5×10^6 – 7×10^6 g/mol polystyrene was reduced by a factor of 2–3.

Concentration Effects. Orvisky et al. (*F3*) studied SEC concentration effects for hyaluronan and found that higher operating temperatures diminished these effects. Fischer and

Siebrands (*F4*) used colloidal CdS with a diameter of 17 nm as a model solute to study the effects of sample concentration on elution volume and peak area. The electric double-layer effect was found to play an important role in elution behavior. Also, a memory effect of the column was observed due to temporarily adsorbed particles. Song and Hu (*F5*) described a theoretical model of concentration effects in SEC.

Viscosity Effects. Plante et al. (*F6*) published an interesting paper on the visualization of viscous fingering in SEC using magnetic resonance imaging (MRI). At bovine serum albumin concentrations greater than 30–50 mg/mL, SEC viscous fingering was observed; however, using MRI, viscous fingering was evident at concentrations as low as 10 mg/mL. The authors concluded that the impact of fingering at lower concentrations is reduced because of dispersion. Similar studies were done with glycerol.

Aqueous SEC Adsorption Effects/Mobile-Phase Selection. Garcia and co-workers published a series of papers on the solution properties of polyelectrolytes. They reported on the elution behavior of several types of polymers as a function of pH and ionic strength on Spherogel TSK PW4000 and Ultrahydrogel 250 (*F7*). Equations were derived from Flory–Fox theory of polymer solutions to explain the adsorption process in terms of preferential interaction (*F8*). They also studied the dependency of eluent ionic strength on elution volume of polyelectrolytes, taking into account both the electric double layer and the effective radius of polyions and assuming both that the solutes behaved as rigid spheres and that the gel pore geometry was cylindrical (*F9*). In further studies, a theoretical treatment was developed to establish the influence of mobile-phase ionic strength on SEC calibration involving the net charge of the polyion and the residual charge on the packing (*F10*).

Volet and Lesec (*F11*) used on-line viscometry and light-scattering detectors to study non-size-exclusion effects of polyelectrolytes. Using an on-line multiangle light-scattering detector and pure water as the mobile phase, Reed (*F12*) found no evidence of flow-induced chain extension during SEC of sulfated glycosaminoglycan in its electrostatically expanded state. Veggeland et al. (*F13*) discussed osmotic effects, caused by the establishment of Donnan membrane equilibrium in the case of polyelectrolytes, when multicomponent eluents were used.

Dubin's group (*F14*) investigated the ionic strength dependency of protein elution on a Superose 12 mixed-mode separation column and introduced a new parameter related to the capacity factor. O'Callaghan et al. (*F15*) reported adsorption of hydrophobic proteins and aromatic amino acids on Superose 12. The effect of mobile-phase composition on the interaction of cephalosporins on Sephadex packings was studied by Changqin and co-workers (*F16*). Corradini et al. (*F17*) examined the salt-mediated elution behavior of proteins on TSK G3000SW. Ovalle (*F18*) separated proteins of similar molecular weight but different *pI* values by nonideal SEC using a zwitterionic buffer to amplify electrostatic interactions between the proteins and silica matrix.

Silvestre et al. (*F19*) evaluated a poly(2-hydroxyethylaspartamide)-coated silica packing for SEC of amino acids and small peptides using a formic acid mobile phase. Weatherell and Lai (*F20*) used a factorial experimental design to examine the interaction of collagen, Croda glue, and gelatin with a polymeric and derivatized silica SEC packing. Rickler et al. (*F21*) used a multivariate visualization mapping technique to study protein–SEC packing interactions. Optimal mobile-phase composition for

ideal SEC could be identified using this approach. Anomalous SEC behavior of notexin and scutoxin, an isoform of notexin, was reported by Francis et al. (*F22*). Multiple late-eluting peaks of these proteins were attributed to association with anions. Hydrophobic interaction between a green fluorescent protein and an SEC packing using high salt concentration was utilized for the purification of this protein (*F23*).

Porsch and Sundeloef (*F24*) studied the SEC and ion exclusion behavior of dextran in water in combination with dynamic light scattering of fractions. Ion exclusion was caused by charged dextran molecules. Bergman et al. (*F25*) investigated the influence of temperature, pH, and ionic strength on the SEC elution behavior of heparin. Shah and Dubin (*F26*) reported on the adsorptive interaction of Ficoll standards with porous glass SEC packings. An explanation given was that sodium ions bind to Ficoll, resulting in the adsorption of the transient pseudopolymerization.

Shaw et al. (*F27*) studied the effect of mobile-phase pH on the SEC of humic acids using Sephadex packings. Kuiters and Mulder (*F28*) investigated the effect of Cu(II) ions in the mobile phase on the elution of humic acids with Sephadex packings. The presence of Cu(II) ions eliminated electrostatic repulsion between solute and gel. DMF was used as the mobile phase for the SEC of trimethylchlorosilane derivatives of humic acids (*F29*).

Using 0.3 M acetic acid and 0.3 M sodium acetate as the mobile phase and Progel-TSK PW as the packing, Swerin and Waagberg (*F30*) characterized cationic polyelectrolytes used in papermaking. SEC of pyrene-labeled polyacrylamides was achieved using 0.2 M sodium sulfate containing 0.05% sodium dodecyl sulfate (SDS) as the mobile phase and μ -Bondagel packing (*F31*). The surfactant was needed to prevent hydrophobic adsorption due to the presence of pyrene in the polymer. Wigman et al. (*F32*) compared an aqueous (water/methanol with Spherogel TSK 2000SW) to a nonaqueous (THF with PLgel column) SEC method for the characterization of ethylene glycol–propylene glycol copolymers and concluded that there was polymer adsorption in the aqueous method.

Nonaqueous SEC Adsorption Effects/Mobile-Phase Selection. A solvent that is being used with increasing frequency for the solubility of “difficult to dissolve” polymers is 1-methyl-2-pyrrolidone (NMP). Johnson et al. (*F33*, *F34*) reported that NMP surpasses THF as an SEC mobile phase for coal tar pitch, in that it prevents adsorption. Masuda et al. (*F35*) also used NMP as the mobile phase with the addition of LiBr for the SEC analysis of preasphaltenes of brown coal heavy liquids and novalak phenolic resins. NMP containing LiBr or purified over P₂O₅ was used with an on-line viscometer for the characterization of polyimides and a polyamic acid precursor (*F36*, *F37*). A DMF mobile phase with and without LiBr and H₃PO₄ was evaluated also for the SEC of polyamide–polyimide and poly(amic acid) (*F38*).

For the characterization of poly(ether ketone), Daoust and co-workers (*F39*, *F40*) first sulfonated PEEK using sulfuric acid and then used NMP containing 0.1 M LiBr at room temperature as the SEC mobile phase. This derivatization procedure and those SEC conditions were used also for the characterization of a semicrystalline poly(aryl ether ketone-*co*-sulfone) (*F41*). Polyether–polyketones were also determined using mixed solvent systems consisting of pentafluorophenol and chloroform (*F42*) and dichloroacetic acid and chloroform (*F43*). A mixed solvent

mobile phase of NMP and toluene containing LiBr and ethylenediamine was used for the SEC of poly(vinylpyridine) (*F44*).

Dimethylformamide, another aprotic solvent, has been used with LiBr for the SEC of sulfonated alternating copolymers of maleic acid and styrene (*F45*) and acrylonitrile polymers (*F46*, *F47*). Ono (*F48*) was awarded a patent for using dimethylacetamide containing LiCl for the SEC of cellulose.

Poly(ethylene terephthalate) and related polymers were analyzed using solvent mixtures of chloroform/hexafluoro-2-propanol (98/2) (*F49*) and methylene chloride/dichloroacetic acid containing 0.01 M tetrabutylammonium acetate (*F50*). Poly(butylene terephthalate) was determined at room temperature using a mixture of *o*-chlorophenol/chloroform (*F51*).

Su et al. (*F52*) evaluated the GPC of polyoxymethylene in hexafluoro-2-propanol (HFIP) in which polymer association was observed. The addition of sodium trifluoroacetate to the mobile phase eliminated this problem. Mori and Nishimura (*F53*) also used sodium trifluoroacetate to HFIP to suppress polyelectrolyte effects during SEC of polyamides. These authors reported that the addition of this electrolyte also changed the elution behavior of poly(methyl methacrylate) used as calibrants. Polyamide 6, prepared by anionic polymerization of 6-hexanolactam, was trifluoroacetylated before SEC analysis (*F54*). However, analysis had to be done within an hour because of hydrolysis (mobile phase not given in abstract). Mourey and Bryan (*F55*) used a methylene chloride/dichloroacetic acid mobile phase for the SEC of different polyamides.

Other unusual mobile phases of interest were methanol/phosphate buffers for poly(methacrylic acid) (*F42*), chloroform/methanol for hydroxypropylmethyl cellulose (*F42*), a metal-amine complex and/or a metal-alkali complex for cellulose and carboxymethyl cellulose (*F56*), and ethanol containing trifluoroacetic acid for an imidazole (*F57*). Guttman et al. (*F58*) reported that the addition of 0.05 M LiBr to THF prevented the occurrence of "concentration-dependent peaks" when analyzing polyurethane. Finally, Mayeda and Nagata (*F59*) compared the use of 1-cyclohexyl-2-pyrrolidinone containing LiCl with 1-chloronaphthalene as mobile phases for the SEC of poly(phenylene sulfide).

DETECTORS

Light-Scattering Detectors. Jackson and Barth reviewed the principles and methodology of molecular weight detection by light scattering and viscometry in conjunction with SEC (*G1*). The use of a multiangle laser light-scattering (MALLS) detector for SEC was evaluated for the characterization of linear and branched polymers by Podzimek (*G2*), Menon et al. (*G3*), and Dayal and Mehta (*G4*). Mourey and Coll evaluated a two-angle light-scattering detector for SEC (*G5*, *G6*) that measures the scattered light intensity at 15° and 90°. The radius of gyration and molecular weight were determined by fitting the ratio of the intensities to some form of the particle-scattering function. Accurate molecular weights were obtained for both linear high molecular weight polystyrenes and branched polyesters. The *z*-average radius of gyration for the whole polymer was determined with high precision and accuracy; however, the values at each elution slice were greatly affected by detector noise.

Cotts (*G7*) used SEC-MALLS to compare the molecular dimensions of dialkyl- and aryl-alkyl-substituted polysilanes. The unperturbed dimensions were estimated by an extrapolation technique using the measured dependence of the radius of

gyration on molecular weight. Dayal and Mehta (*G8*, *G9*) used MALLS and high-temperature SEC to characterize molecular weight and branching in polyethylene. Pille and Solomon (*G10*) used MALLS to study the formation of microgel during the cross-linking of living poly(4-*tert*-butylstyrene) by 1,4-divinylbenzene. They found that primary particles are formed early in the reaction and appear to have a comblike structure. These primary particles undergo interparticular cross-link reactions to form a network. The networks themselves can also undergo further intraparticular cross-link reactions.

Low-angle laser light scattering (LALLS) and ¹³C-NMR were used by Minagawa et al. (*G11*) to study the molecular weight distribution and tacticity of polyacrylonitrile prepared by urea clathrate polymerization. Grubisic-Gallot and co-workers (*G12*) studied the influence of copolymer composition and molecular weight on polystyrene-*block*-poly(methyl methacrylate) micelles in the mixed solvent 1,4-dioxane/cyclohexane.

Mourey and Balke (*E20*) proposed a simple method for using SEC with both concentration and light-scattering detectors to calibrate other chromatographs having only concentration detectors. Wyatt and Papazian (*G13*) discussed the determination of the interdetector volume. Shortt (*G14*) showed that MALLS measurements of the radius of gyration could be used to estimate the polydispersity of narrow-distribution polymers independent of the interdetector volume. SEC-LALLS was used to compare the degradation of high molecular weight polystyrenes on different column sets (*G15*). SEC-MALLS was used to study the kinetics of acid hydrolysis (*G16*) and to study the helix-coil transition (*G17*) in carrageenans.

SEC light scattering was also used to characterize pectins (*G18*), glycoproteins (*G19*), propellant binders (*G20*), polyurethane elastomers (*G21*), dimethylamine-epichlorohydrin copolymers (*G22*, *G23*), unsaturated polyester resins (*G24*, *G25*), nitrocellulose (*G26*), poly(vinyl alcohol) (*G27*), and polyamide copolymers (*G28*).

MALLS was also used in conjunction with thermal (*N13*, *N14*), sedimentation (*N19*), and flow (*N17*) field-flow fractionation. These papers are discussed in section N on field-flow fractionation.

Viscometers. Lesec (*G29*) reported on flow rate fluctuations in capillary viscometers, in which the fluctuation is caused by the change in pressure as the polymer peak flows through the capillary. Ways to minimize flow rate fluctuations were discussed. Lew et al. (*G30*) reviewed the use of SEC viscometry for high-temperature analysis of polyolefins. Lesec et al. (*G31*) described the use of a single-capillary viscometer with high-temperature SEC for the analysis of polyethylene. Balke et al. (*G32*) evaluated different methods of determining the number-average molecular weight using SEC viscometry. They found that methods based only on the viscometer response and the universal calibration curve were 2–3 times less precise than conventional universal calibration methods. They also examined sources of error in the determination of number-average molecular weight.

Harrison and Mourey (*G33*) used SEC viscometry with refractive index and UV detectors to measure the number-average molecular weight and functionality of poly(tetramethylene glycol). The hydroxyl groups were derivatized to provide an end-group-selective UV-absorbing tag. The number of end groups per chain could be calculated from the chromatogram using the UV detector. This approach was used to evaluate the accuracy and limitations

of different methods of calculating the molecular weight of low-molecular-weight polymers.

Timpa and co-workers (*G34–G37*) used SEC viscometry in conjunction with a dimethylacetamide and lithium chloride mobile phase to characterize starches and cellulose. Siochi et al. (*G38, G39*) characterized the molecular weight distribution of poly-(amide acid) with SEC viscometry. Xie (*G40*) used SEC viscometry to determine the Mark–Houwink–Sakurada coefficients for polystyrene and polyisobutene.

Combined Light Scattering/Viscometry. A number of papers discussed data treatment and interpretation in SEC with combined light scattering and viscometry. Reed (*G41*) presented a quantitative investigation of systematic and random sources of error in multidetector SEC and their influence on polymer characterization. The emphasis was on aqueous SEC, but the principles were expected to carry over to organic-phase SEC. Rudin and co-workers (*G42, G43*) presented new methods for correcting for the interdetector volume in multidetector SEC using independent calibration curves of hydrodynamic volume versus elution volume for each detector. Netopilik (*G44*) and Jackson and Yau (*G45*) presented model analyses of the characterization of linear polymers using multidetector SEC. Characterization of the molecular weight distributions of branched polymers was also studied (*G46*). Bruna (*G47*) reviewed multidetector SEC, and Jackson and Barth reviewed some of the inherent problems in multidetector data analysis (*G48, G49*).

Degoulet and co-workers (*G50*) used multidetector SEC to characterize polydisperse solutions of branched poly(methyl methacrylate). They used a new type of multiangle light-scattering detector to determine the static structure factor and the mass distribution. These were found to be consistent with the predictions of percolation theory. Lesec et al. (*G51*) studied star-branched polymers and found excellent agreement between results from light scattering and from universal calibration, even for highly branched stars. Wintermantel et al. (*G52*) studied linear and highly branched polystyrenes and observed peculiar elution behavior for highly branched microgels. Zigon and colleagues (*G53*) studied polyether–polyurethanes and found deviations between light scattering and universal calibration results above 60 000 g/mol, due to the relatively high intrinsic viscosities of the polyurethanes.

Multidetector SEC was also used to characterize poly(phenyl-acetylenes) (*G54*), carbohydrate biological response modifiers (krestin, schizophyllan, and glucan phosphate) (*G55*), poly(4-vinylpyridine) (*G56*), dextrans and pullulans (*G57, G58*), and copolymers of vinylpyrrolidone and 2-methyl-5-vinylpyridine (*I57*).

Chemiluminescent Nitrogen Detection. Fujinari and Manes (*G59*) coupled this detector to an SEC column for the characterization of peptides and food-grade hydrolyzed proteins.

Density Detector. Trathnigg's group (*G60–G63*) has been actively involved with the use of on-line density detection. This detector has been applied to the analysis of oligomers (ethoxylated fatty alcohols and ethylene oxide–propylene oxide block copolymers) using liquid chromatography under critical conditions coupled to SEC columns. Using a combination of density and refractive index detection, these workers were able to characterize oligomers in terms of molecular weight, chemical composition, and functionality.

Dynamic Surface Tension. Synovec's group (*G64*) described the design and evaluation of a dynamic surface tension

detector that measures optically the drops at the end of a capillary tube connected to the outlet of the column. Surface-active solutes cause a significant decrease in the drop volume that can be measured readily. The application of this device for detecting trace high molecular weight impurities in high-concentration, low molecular weight polymer matrixes was discussed.

Evaporative Light Scattering. An evaporative light-scattering detector (ELSD) was used for the SEC of edible oils (*G65*) and amide-type detergents in gasoline (*G66*). Rissler and co-workers (*G67–G70*) employed an ELSD for the analysis of poly-(ethylene glycol) (*G67*), poly(butylene glycols) (*G68, G69*), poly(propylene glycol) (*G69*), and acetylated poly(propylene glycol) di- and triamine (*G70*) oligomers by normal- and reversed-phase HPLC. Using an ELSD, Kawai et al. (*G71*) analyzed poly-(methyl methacrylate)-*graft*-poly(dimethyl siloxane) using reversed-phase HPLC.

Flame Ionization. An ultrahigh-temperature SEC system was developed by Qian and Nagashima (*G72*) for determining the molecular weight distribution of poly(phenylene sulfide), poly-(ether ketone), poly(ethylene terephthalate), and polyolefins. Using 1-chloronaphthalene as the mobile phase at 210 °C, these authors used either a UV viscometer or a flame ionization detector–viscometer combination.

Fourier Transform Infrared. Cheung et al. (*G73, G74*) developed and evaluated two types of FT-IR detector interfaces which removed solvent prior to FT-IR spectrophotometric measurements. The morphology of the deposited polymer film was found to affect spectra. This group (*G75*) also investigated factors affecting the quantitative analysis of polymer composition distribution by SEC using a solvent evaporation interface for FT-IR. A commercially available FT-IR interface was used as an SEC detector by Willis and Wheeler (*G76*), Provder et al. (*G77*), and van Every and Long (*G78*). Yamamoto et al. (*G79*) used an on-line FT-IR detector to examine the molecular weight dependency of the poly(oxyethylene) content of dimethylsiloxane–methylpoly-(oxyethylene)siloxane copolymers.

High Frequency. A high-frequency detector for SEC of polymers and colloids was reported by Faimon and Ondracek (*G80*).

Inductively Coupled Plasma Mass Spectrometry. SEC-ICPMS was used to monitor metal content in the stomach and intestinal contents of guinea pigs (*G81–G83*) and selenium-containing proteins in human serum (*G84*).

Mass Spectrometry. A review (in Chinese) by Long and Lu (*G85*) discussed developments and applications of electrospray ionization of biopolymers using HPLC-MS and SEC-MS. Simonsick and Prokai (*G86*) used electrospray ionization MS as an on-line SEC detector for the characterization of ethylene oxide-based surfactants, acrylic macromonomers, and waterborne clearcoat formulations.

Montaudo and co-workers (*G87, G88*) used matrix-assisted laser desorption/ionization (MALDI) time-of-flight (TOF) MS to characterize SEC fractions of poly(butylene adipate) and poly-(butylene adipate-*co*-butylene succinate) (*G87*) and poly(dimethylsiloxane) (*G88*). Lloyd et al. (*G89*) found good agreement between MALDI-TOFMS and SEC data for molecular weight averages for narrow polydispersity poly(methyl methacrylates) (PMMA) and polystyrene (1000–12 000). However, a study by Lehre and Sarson (*G90*) reported significant molecular weight differences for PMMA obtained from SEC and MALDI-

MS. Finally, Herod and Kandiyoti (*G91*) found qualitative agreement for high-molecular-mass materials in coal-derived liquids using SEC, MALDI, and laser ionization MS techniques.

Postcolumn Detection. A Superose column was used to characterize organically bound Al in natural water samples using a postcolumn reaction with pyrocatechol violet (*G92*).

PACKINGS

Inorganic-Based Packings. In-depth coverage of silica-based SEC columns was presented by Eksteen and Pardue (*H1*). Petro et al. (*H2*) synthesized and evaluated dextran-grafted silica packings for SEC of proteins. Porsch (*H3*) studied bonding reactions of glycidoxypolytrimethoxysilane with silica, which were evaluated with poly(ethylene glycols).

Polymeric-Based Packings. Meehan (*H4*) presented a comprehensive review of polymeric-based SEC packings. A patent on porous packings made from copolymers of siloxanes was awarded to Nozu (*H5*). Petro et al. (*H6*) described composite packings formed by silica, poly(hydroxyethyl methacrylate), or carbon-based macroporous particles with pores filled with a dextran network.

Smigol and co-workers (*H7*) synthesized and tested poly(4-hydroxystyrene-*co*-divinylbenzene) SEC packings. Kim et al. (*H8*) prepared and evaluated monodisperse porous poly(styrene-*co*-divinylbenzene) gels and poly(acrylonitrile-*co*-ethylene dimethacrylate) gels. Wang et al. (*H9*) controlled the pore structure of monodisperse macroporous particles of poly(styrene-*co*-divinylbenzene) using linear polystyrene as a porogen. Li et al. (*H10*) produced packings based on a cross-linker having independent vinyl groups, e.g., 1,2-bis(*p*-vinylphenyl)ethane and its meta isomers were suspension copolymerized with *p*-methylstyrene in the presence of different porogens.

The SEC properties of poly(vinyl alcohol) particles prepared by a freezing-and-thawing procedure were investigated by Murakami et al. (*H11*). Di-, tri-, and oligo(ethylene glycol) dimethacrylates were prepared and evaluated for aqueous SEC by Ogino et al. (*H12*). The characteristics of Superose 12 for protein separations was studied by Dale et al. (*H13*). To minimize shear degradation, Shioda and co-workers (*H14*) evaluated UT-800 series columns, which consist of larger size particles, for high-temperature SEC of polyethylene.

A patent was issued for packings prepared by grafting polymer beads with a monomer, such as glycidyl methacrylate, cross-linking the product by ether formation between epoxide groups, and converting unreacted epoxides to hydroxyl groups (*H15*). Hara et al. (*H16*) were granted a patent for a cellulose-based packing for aqueous SEC.

COMPOSITIONAL HETEROGENEITY

The determination of compositional or chemical heterogeneity of macromolecules as a function of molecular weight is an important area of research. In this section, various separation approaches are covered: SEC with selective detectors, interactive HPLC, which includes separation by sorption and/or solubility and "critical condition" chromatography, temperature-rising elution fractionation, supercritical fluid extraction and chromatography, and classical fractionation. For related articles on determining compositional heterogeneity, please consult sections G, M, and N respectively on Detectors (mainly FT-IR and mass spec-

trometry), Coupled Columns/Column Switching, and Field-Flow Fractionation.

SEC with Selective Detectors. Barth (*B12*) reviewed recent developments in the use of hyphenated multidimensional separation and detection techniques for polymer characterization, including determining compositional heterogeneity using SEC with on-line selective detectors. Huber (*I1*) covered the use of UV-visible diode array detection for the analysis of polymer products. The use of SEC with UV detection for analyzing telechelic and functional polymers was given by Priddy et al. (*I2*). Thermal discoloration of styrene-*co*-acrylonitrile polymers was investigated using SEC/UV-visible detection (*I3*, *I4*). Utilizing SEC with a UV detector and refractometer, Huang and Sundberg (*I5*) determined grafting efficiency of styrene onto *cis*-polybutadiene.

Bielsa et al. (*I6*) emphasized data treatment problems associated with the estimation of the independent distributions of molecular weight and functionality of hydroxyl-terminated polybutadiene employing SEC with a UV refractometer detection system. Calculations are presented that include absolute detector calibrations and corrections for the dependency of molecular weight on refractive index and instrumental band broadening. Paniker and Ninan (*I7*) used the dual-detector approach for estimating the functionality distribution of the extractable sol from hydroxy-terminated polybutadiene after curing with toluene diisocyanate. These authors (*I8*) also used adsorption LC to fractionate 3,5-dinitrobenzoyl esters of hydroxy-terminated polybutadiene, which were then analyzed by SEC with dual detection.

Meehan and O'Donohue (*I9*) reported on the use of a low-angle light-scattering detector in series with a UV detector and refractometer for measuring the molecular weight distribution and chemical composition distribution of polystyrene-*block*-poly(methyl methacrylate) and polystyrene-*block*-poly(ethylene oxide). Lee et al. (*I10*) characterized mixtures of polystyrene and poly(methyl methacrylate) using SEC with dual detectors. Holland et al. (*I11*) investigated the pyrolysis of a substituted poly(cyclohexadiene) into poly(*p*-phenylene), in which phenylene residues were monitored as a function of molecular weight.

Interactive HPLC. Lochmueller et al. (*I12*) reviewed strategies for predicting retention in HPLC, including retention models for polymers. Mori (*I13*, *I14*) presented a comprehensive review on the use on interactive HPLC for the characterization of copolymers. This researcher also studied the elution behavior of styrene-acrylonitrile copolymers using isocratic reversed-phase HPLC with chloroform/hexane mixtures as mobile phases (*I15*, *I16*). Ogino (*I17*) reviewed the use of solvent gradient HPLC to characterize methyl methacrylate-styrene copolymers and the stereoregularity of poly(methyl methacrylate) and to determine the isomeric structure of polybutadiene. Ogino and co-workers (*I18*) evaluated the use of acrylonitrile, acrylamide, polystyrene, and octadecyl methacrylate HPLC packings to determine the chemical composition of styrene-*n*-butyl methacrylate and styrene-*tert*-butyl methacrylate copolymers.

Gloeckner et al. (*I19*–*I22*) published a series of papers on the control of adsorption and solubility in gradient HPLC of styrene-ethyl methacrylate, styrene-methyl methacrylate, and styrene-acrylonitrile using either normal- or reversed-phase systems. Schunk (*I23*) used normal-phase HPLC to determine the composition distribution of methyl methacrylate-methacrylic acid copolymers followed by SEC to obtain a chemical composition/molecular weight map of these copolymers. Tanaka et al.

(*I24*) determined the chemical composition distribution of poly-(methyl methacrylate)-*graft*-polystyrene using normal- and reversed-phase HPLC.

Pattueli et al. (*I25*) measured the sequence distribution of styrene-butadiene copolymers using reversed-phase HPLC with a ternary gradient and an ELSD. Okamura and Nagata (*I26*) used normal-phase LC to determine the chemical composition distribution of vinyl chloride-*N*-cyclohexylmaleimide copolymers. Frechet and co-workers (*I27*) separated functionalized dendritic macromolecules with reversed-phase HPLC. Polyoxometalates, polymers of transition metal oxides, were analyzed using HPLC and capillary zone electrophoresis (*I28*).

Critical retention behavior of polystyrene and polybutadiene was studied by Cools and co-workers (*I29*). Under critical conditions, the retention of a polymer is independent of its molecular weight and depends only on its chemical composition. This group also proposed an approach for determining critical conditions. Polyesters were characterized by Drueger and colleagues (*I30*) using liquid adsorption chromatography at critical conditions in which the polyesters were separated according to their end groups. Furthermore, cyclic species, hexanediol ether structures, and alkyl-terminated homologues were identified. Fractions were analyzed using off-line SEC and MALDI-TOFMS.

Monomers, additives, oligomers, and polymers were extracted using a multisolvent gradient approach with a guard column (*I31*). Bullock (*I32*) evaluated a high-temperature HPLC method using nonporous reversed-phase packings for characterizing a polymeric contrast agent. Over 50 individual oligomer peaks were resolved. Selected examples of oligomeric analysis using HPLC are bisphenol A dicyanate oligomers (*I33*), nonylphenol ethylene oxide oligomers (*I34*, *I35*), nylon oligomers (*I36*), perfluoropoly(oxyalkylene) oligomers (*I37*), poly(butylene glycol adipate) oligomers (*I38*), poly(ether sulfone) oligomers (*I39*), vinyl chloride oligomers (*I40*), and tributylphenol ethylene oxide oligomers (*I41*).

Supercritical Fluid Chromatography/Extraction. Ude (*I42*) presented a review on the fractionation of polymers with supercritical fluid chromatography (SFC). McHugh et al. (*I43*) discussed the use of supercritical fluid extraction (SFE) in polymer fractionation and provided a thermodynamic basis to aid in choosing an appropriate SFE solvent. Urata and Nitta (*I44*) used SFC with CO₂ or ethylene with THF to separate styrene-maleic anhydride copolymers and presented an equilibrium desorption model to simulate elution behavior. SFE was used to prepare monodisperse polystyrene using supercritical THF (*I45*) and low-polydispersity maleic anhydride copolymers with supercritical CO₂ (*I46*).

Ute et al. (*I19*) used preparative SFC to fractionate oligomers of methyl methacrylate with CO₂/ethanol as the mobile phase on silica. Capillary column SFC and HPLC were used to separate nonionic octylphenol polyether alcohols (*I47*) and polyethoxylated nonylphenol surfactants (*I48*). Polar oligomers that were found in the former samples using HPLC were not detected by SFC because of their insolubility in supercritical CO₂ (*I47*). A patent was awarded to Lin and Chen (*I49*) for fractionating poly-(tetramethylene ether glycol) using propane with THF as the cosolvent.

Solvent Fractionation. Monrabal (*I50*) developed a new technique for analyzing short-chain branching distribution in linear low-density polyethylene. The technique, referred to as crystallization analysis fractionation, is based on a stepwise precipitation

approach by which polymer concentration is monitored during crystallization. Mencer and Gomzi (*I51*) developed a model for column fractionation of polymers in which the concentration profiles of polymer fractions can be computed and predicted. Methot (*I52*) discussed the use of various methods including differential scanning calorimetry (DSC) to fractionate polyolefins based on molecular weight and short-chain branching. Koivumaki and Seppala (*I53*) used LC to fractionate ethylene-propylene copolymers as a function of their compositions.

Isotactic polypropylenes were fractionated using Soxhlet extraction methods, and results were compared to those of solvent-nonsolvent methods (*I54*). The technique of foam fractionation was reported by Twite et al. (*I55*) for the characterization of water-reducible acrylic coating systems.

Cross-Fractionation (also see section M). Preparative cross-fractionation of methyl methacrylate-methacrylic acid copolymers was achieved using solvent-nonsolvent and temperature gradient fractionation procedures (*I56*). Cross-fractionation of copolymers of *N*-vinylpyrrolidone and 2-methyl-5-vinylpyridine was determined by first fractionating according to molecular weight by SEC and then by composition using reversed-phase HPLC (*I57*).

Temperature-Rising Elution Fractionation. Wild and Blatz (*I58*) described a high-performance temperature-rising elution fractionation (TREF) system for the separation of polyolefins based on crystallinity. Borrajo et al. (*I59*) applied a simple thermodynamic model, based on an extension of the Flory-Huggins theory, to TREF. This model was used to predict the dependency of the fractionation process on melting temperature, melting enthalpy, average crystallinity, average crystallinity sequence length, and polymer-solvent interaction parameter. Results from the model also predicted number-average branch points of TREF fractions.

Mirabella (*I60*) used preparative TREF to fractionate a propylene-ethylene copolymer. The fractions, which were characterized by ¹³C-NMR to determine ethylene content, were used as standards to calibrate an analytical TREF system. Rudin and Pigeon (*I61*) generated a TREF calibration curve based on the relationship between branch frequency and elution temperature of polyethylene by using a dual-IR detection system which measures both concentration and branching frequency.

Soares and Hamielec (*I62*) presented an overview of TREF and discussed TREF/¹³C-NMR measurements of ethylene-propylene copolymers and polypropylenes synthesized using metallocene catalysts as well as heterogeneous Ziegler-Natta catalysts. This group (*I63*) analyzed TREF data of polyolefins, prepared from Ziegler-Natta catalysts using the Stockmayer bivariate distribution. Parker et al. (*I64*) reported that TREF of linear low-density polyethylenes prepared from a metallocene catalyst gave a narrow distribution, as compared to the broad distribution obtained from polyolefins polymerized using Ziegler-Natta catalysts. Using TREF, DSC, and ¹³C-NMR measurements on propene-*co*-butene random polymers polymerized using Ziegler-Natta catalysts, Collina et al. (*I65*) reported that the copolymers are blends of nearly Bernoullian macromolecules having different compositions.

Fernandez et al. (*I66*, *I67*) employed TREF to fractionate a series of linear low-density polyethylene samples and obtain their comonomer distribution. Lee and Kim (*I68*) examined the structural and thermal properties of TREF fractions of very low-density polyethylene. TREF was used to help characterize

ethylene–octene linear low-density polyethylenes that were reactively extruded with low levels of peroxide (I69). Yamamoto and co-workers (I70) characterized the composition of resins used for injection molding. Various copolymers of ethylene and α -olefins were analyzed by TREF, SEC, and ^{13}C -NMR (I71). Usami et al. (I72) described a TREF-SEC coupled system for determining the chemical composition distribution of impact-resistant propylene–ethylene copolymers.

Keating and McCord (I73) described a DSC procedure for fractionating ethylene copolymers on the basis of crystallite size, which is related to ethylene block length. The technique is somewhat similar to TREF, but the fractionation takes place in the bulk phase and there is no physical separation. Mara and Menard (I74, I75) compared TREF to DSC using linear low-density polyethylene as an example. Both techniques gave similar composition–distribution information.

PHYSICOCHEMICAL STUDIES

Synthetic Polymers. *Branching/Mark–Houwink–Sakurada Coefficients/Conformation.* Conventional single-detector SEC techniques were used by Tsitsilianis and Ktoridis (E17) to determine branching in star polymers. Mark–Houwink coefficients for poly(octadecyl methacrylate) (J1), polyisobutene (G40) and vinylidene chloride–methyl acrylate copolymers (J2) were determined using conventional SEC. Papers describing the characterization of branched polymers using molecular weight-sensitive detectors are described in section G of this review.

Specific Refractive Index. The specific refractive index increment of water-soluble polymers in pure water was determined by Bo et al. (J3) using SEC and a differential refractometer. With an electrolyte-containing mobile phase, the measurement is similar to a dialysis experiment in which a constant chemical potential is established. By measuring the specific refractive index of oxyethylene–oxypropylene copolymers of different end-groups, Wang et al. (J4) proposed a method for estimating simultaneously the molecular weight and composition of short-chain copolymers using SEC. Lee and Chang (J5) used an internal standard method to determine specific refractive index increments of polystyrene, poly(styrene-*stat*-acrylonitrile), poly(dimethylsiloxane), and poly(styrene-*stat*-methyl methacrylate) in THF, toluene, and chloroform.

Association/Complexation/Solvation Studies. Grubisic-Gallot and co-workers (J6) used SEC to study the micellar system polystyrene-*block*-poly(methyl methacrylate) in the mixed solvent 1,4-dioxane/cyclohexane. Using an on-line low-angle light-scattering detector, molecular weights of micelles were determined. The influences of flow rate and copolymer concentration were examined. Quintana et al. (J7) employed SEC to determine the association equilibrium of polystyrene-*block*-poly(ethylene propylene) and polystyrene-*block*-poly(ethylene butylene)-*block*-polystyrene copolymers as a function of temperature.

SEC was used by Bu et al. (J8) to study the micellization behavior of a series of monodisperse polystyrene–polyisoprene diblock copolymers. Bhalekar and Engberts (J9) reported on the elution behavior of solutes in the presence of micellar and premicellar aggregates of micelle-forming surfactants in Sephadex G-25 columns. Andersen (J10) determined the effect of concentration on the elution profile of petroleum asphaltenes for measuring critical micelle concentrations and microstructure.

Fischer et al. (J11) proposed an SEC procedure for determining the complexation of macromolecules with low molecular weight solutes, such as preferential solvation. The method is based on the assessment of system peaks formed as a result of differences between eluent composition and bulk solvent composition due to the complexation within the initial sample solution. SEC was used by Porcar and colleagues (J12) to investigate the preferential interactions of polymers with low molecular weight solutes to determine a preferential solvation parameter of ternary polymer solutions. The system studied was poly(vinylpyrrolidone) and poly(4-vinylpyridine) in binary aqueous solutions of nicotine or silicic acid. Trathnigg and Yan (J13) used SEC with a density detector and differential refractometer to measure preferential solvation of poly(oxyethylenes) with hydroxy and methoxy end-groups in chloroform containing ethanol. A method is described for determining adsorbed ethanol molecules per repeating unit and per end group.

Veggeland et al. (J14, J15) studied surfactant–polymer interactions with SEC with and without electrolyte. The method is based on the dynamic dialysis of injected polymer, using the surfactant in the mobile phase. The surfactant–polymer complex is separated from the vacant surfactant solution, and the vacant peak, which is related to the amount of surfactant complexed with the polymer, is quantified using a refractive index detector. This method also describes the amount of salt associated with the charged complex, the Donnan effect. Draper et al. (J16) described an SEC approach for determining the solubilization of drugs in micellar systems. The drug is injected into a mobile phase which consists of the surfactant, and the association equilibrium of the eluent is probed. The surfactant was a triblock copolymer composed of oxyethylene and oxypropylene.

Tian and Tan (J17) determined the miscibility limit of ethylene oxide–THF copolymer with nitroglycerol and 1,2,4-butanetriol trinitrate. Self-organization of a supramolecular aggregate was studied using SEC by Mathias et al. (J18). Stereocomplex formation between isotactic and syndiotactic pentacontamers of methyl methacrylate was observed from SEC analysis (J19). Warwick et al. (J20) examined nickel complexation with fulvic acid in groundwater with SEC.

Kinetic Studies. Kissin (J21) described equations for analyzing SEC distributions to obtain polymerization kinetic data and to study the behavior of active catalyst centers in polymerization reactions. Guillot and Leroux (J22) used SEC data to model the molecular weight distribution and rates of polymerization regarding the grafting of polymers onto silica. Thompson et al. (J23) studied the kinetics of formation of polyurethane prepolymers, which involved quenching of the isocyanate, separation of the oligomeric species by SEC, and measurement by UV absorbance of the quenched moieties. Di and co-workers (J24) used aqueous SEC to determine the kinetics of a water-in-water emulsion polymerization of acrylamide in a poly(ethylene glycol) solution.

Nguyen (J25) reviewed the use of SEC to study mechanochemical degradation kinetics of polymers. Florea (J26) reported a new SEC method for measuring the kinetics of mechanical degradation of polymers in solution. The procedure is based on the facts that mechanical degradation is a first-order reaction and that macromolecules are cleaved mostly in their central region.

Kadivec et al. (J27) described the use of SEC to study the efficiency of peptization agents to reduce the molecular weight of natural rubber during mastication. Results showed that chain

Table 1. SEC Applications of Biochemical Interest

target	matrix	purpose ^a	refs
Proteins, Peptides, and Conjugates			
band 3 protein	crude solubilized and purified	P	J41, J42
solubilized rat liver membrane proteins	rat liver plasma membrane	I, P	J43
porin	bacterial outer membrane	P	J44
MIP P25	insect epithelial membranes	P	J45
gap junction connexons	solubilized rat liver membranes	P	J46
cGMP-dependent protein kinase	purified	P	J49
clathrin proteins	purified	P	J50
plasminogen and fragments	purified	P	J52
rat phenylalanine hydroxylase	purified	P	J53
immunoglobulins	purified, crude, various	I, P, A	O95–O98, J56, J66, J67
hepatitis B surface antigen	purified	A	O99
chaperonins	various states, species	I, P, A	J56–J63
avian sarcoma virus integrase	purified	P	J69
guanylyl cyclase	purified	P	J70
G-proteins	various	P	J71, J78
tropoinins	purified	P	J72
formiminotransferase cyclodeaminase	purified	P	J73
voltage-dependent K ⁺ channel	in vitro translation products	P	J74
actin	purified	P, A	J75
factor IX and domains	purified	P	J76, O102
gp41	purified	P	J79
galectin	purified	P	J80
HIV reverse transcriptase	purified	P	J82
apoferritin	purified	P	J83
serine hydroxymethyltransferase	purified	P	J87
trp aporepressor	purified	P	J89
porcine glutathione S-transferase	purified	P	J90
superoxide dismutase	purified	P	J91
interleukin 4	purified	P	J94
basic fibroblast growth factor	purified	P, A	J96, J97
insulin and dextran conjugates	purified	P, A	O100, O101
somatotropin	purified	A	O103, O104
rhGH	purified	A	O105–O109
Na ⁺ /K ⁺ pump	purified and tissue homogenates	P, A	O110
milk proteins	various	A	O114, O115
wheat proteins	various extracts	P, A	O116–O119
soybean proteins	various	A	O120, O121
muscle cell culture medium	culture medium	P, A	O125
peptides	various	P, A	J51, J68, J77, J92, O122–O124
Nucleic Acids			
sheared DNA and duplex polynucleotides	purified	P	J54
plasmid	purified	I	O128
various RNAs and DNAs	review	I, P, A	O127
degraded DNA	crude tissue extracts	A	O129
Others			
hyaluronate	purified	P, A	J55, O134
pectins	various	A	O131, O132
lipids	various	I, P, A	O135, O136

^a I, isolation or purification; A, assay or activity determination; P, physical characterization.

scission in the mastication process, with or without peptization agents, is a random process.

Inverse SEC. Inverse SEC (or HPLC) is a method in which a material of interest is packed into a chromatographic column, and solutes are injected to probe either the porosity or surface characteristics of the packing. Revillon (*J28*) reviewed the applications of inverse SEC for porosity measurements. Sernetz (*J29*) used SEC to determine the fractal structure in swollen gels. In a swollen gel, the border between the solid phase (polymer network) and liquid phase (solvent) can be characterized as a fractal surface. The fractal dimension of this border can be determined from the cumulative exclusion volume as a function of pore size.

Hayashi et al. (*J30, J31*) used inverse LC to evaluate the interaction between solvent-swollen coal and aromatic solutes and to determine the porous structure of coals. Calcium alginate particles were characterized with proteins of known Stokes radii (*J32*). Inverse SEC was used to characterize cellulosic hollow-

fiber hemodialysis membranes (*J33*) and water-swollen rayon and cotton fibers (*J34*).

Biopolymers. Structure/Conformation Studies. Estimation of the molecular mass and/or size of native and recombinant proteins, synthetic peptides, and complexes of proteins by SEC has become a standard method. Protein chemists have taken advantage of the rapid and convenient determinations that can be conducted using small-particle, high-resolution HPLC columns. Examples of the estimation of protein molecular mass or volume are included in the section on Selected Applications and also appear in Table 1. Examination of these studies illustrates the increasing range of experimental conditions which may be used for SEC analyses.

A brief description of laboratory conditions for estimating native protein molecular weight by SEC was presented by Irvine (*J35*). Himmel et al. (*J36*) presented a very clear view of the issues that must be considered for SEC column calibration for determination of protein molecular weight or hydrated radius. Harlan et al. (*J37*)

described the use of a double-Gaussian distribution for column calibration of the SEC distribution coefficient with Stokes radius for a broad range of globular proteins.

Analysis of the subunit structure of native proteins and enzymes can be accomplished by SEC determination of apparent molecular weight under native conditions, using the assumption of a globular protein structure for column molecular weight calibration. This determination is followed by analysis of molecular mass using denaturing conditions to determine the oligomerization state of the native molecule. The determination of dissociated (denatured) subunit molecular mass can be achieved by SEC, e.g., with a mobile phase containing 6–8 M guanidine-HCl (GuHCl), 6–8 M urea, or 0.05–0.2% sodium dodecylsulfate (SDS), or by using SDS–polyacrylamide gel electrophoresis (SDS–PAGE) or mass spectral analysis of selected peaks. There were many reports of this approach during the review period for the investigation of soluble cytosolic and secreted proteins; selected examples include bovine kidney protein phosphatase 2A inhibitors (*J38*), bacterial DsbC protein (*J39*), and worm extracellular hemoglobin (*J40*). This approach has also become valuable for the analysis of the subunit structure of membrane proteins. Examples of the determination of membrane protein subunit structure include native and mutant recombinant human plasma membrane anion-exchange proteins, i.e., band 3 proteins (*J41*, *J42*). The useful combination of SEC under native and denaturing conditions with chemical cross-linking, using the bifunctional reagent dithiobis(succinimidyl propionate) and SDS–PAGE, was described for the subunit analysis of solubilized rat liver membrane proteins, including integrin heterodimer, dipeptidyl aminopeptidase IV, and cell-CAM 105 (*J43*). The combination of SEC and LALLS was successfully applied to the study of the oligomerization state of membrane porin (OmpF), which had been solubilized from bacterial outer membrane (*J44*).

A number of studies employed SEC determination of the Stokes radius both as a hydrodynamic measurement of particle volume and to assess the effects of experimental manipulations on protein conformation. A reasonably complete hydrodynamic description of a protein can be obtained by combining the Stokes radius (commonly determined by SEC) with the sedimentation coefficient (from ultracentrifugation) and/or the diffusion coefficient (from light scattering) and intrinsic viscosity. The combination of these measurements permits a more accurate estimation of molecular weight (and probable subunit composition) and definition of particle asymmetry. Selected studies of proteins include the insect epithelial membrane major intrinsic protein P25 (MIP P25) (*J45*), rat liver gap junction membrane connexons (*J46*), recombinant bovine calmodulin-stimulated phosphodiesterase PDE1A2 and a deletion mutant, Δ M5-PDE1, (*J47*), a novel nuclear transcriptional factor from cultured keratinocytes (*J48*), native cGMP-dependent protein kinase (cGK) I α and recombinant cGK II (*J49*), and the β 2 chain of the clathrin-associated protein complexes (*J50*).

SEC analysis was applied both to the investigation of environmental effects on the aggregation state and conformation of a 20-residue synthetic peptide corresponding to internal sequences of hen lysozyme (*J51*) and to the conformational changes that result from the binding of 6-AHA and t-AMCHA to human plasminogen and defined fragments of the protein (*J52*). The distinction between resting and activated states of recombinant rat hepatic phenylalanine hydroxylase was apparent from measurement of

hydrodynamic volumes, as well as in the tendency of each of the homotetramers to disaggregate to dimers in response to changes in solution conditions (*J53*).

SEC-LALLS was applied to the analysis of ultrasonically sheared DNA and various polyribonucleotide heteroduplexes (*J54*). Defined fractions of the nucleic acids were isolated by selective acetone precipitation and characterized for weight-average molecular weight and polydispersity. The Mark–Houwink relationships were evaluated for each of the various polyribonucleotide duplexes and were found to vary depending on the conformation of the helices.

The Stokes radii of sodium hyaluronate fractions were studied by SEC combined with intrinsic viscosity determinations (*J55*) to define the useful range of separations on Sephacryl S-1000 and to define the conformation of high molecular weight fractions.

Associations/Protein Folding. As was apparent in the previous review period, the largest growth in biochemical SEC analyses during the last 2 years has been in characterizing protein and peptide folding, association, and aggregation kinetics. These topics are intimately related, especially in cases where the studies address folding patterns of multisubunit proteins, where monomeric proteins exhibit a folding intermediate which passes through a self-associated state, or when aggregation may involve a partly unfolded polypeptide. The driving force for studies in this area include the abundant availability of proteins expressed by recombinant DNA techniques and of smaller protein fragments generated by improved peptide synthesis, as well as improved knowledge of the rules governing protein folding and subunit associations.

The chaperonins are a series of proteins which assist in the normal in vivo processes of nascent polypeptide translocation, folding, and assembly. SEC has been used heavily to study chaperonin polypeptide structure and conformation, associations with other chaperonins, and associations between chaperonins and unfolded or partially folded substrate polypeptides. The relatively high stability of some chaperonin–substrate complexes permits quantitation of binding stoichiometry and particle hydrodynamic description. Studies using SEC for the hydrodynamic analysis and isolation of chaperonin complexes include bacterial and yeast Hsp90 dimers and oligomers analyzed at various concentrations of guanidine and divalent cations (*J56*); the chaperonin 10 aggregates from human, *Escherichia coli*, and *Mycobacterium tuberculosis* (*J57*), studied at various protein concentrations, with or without added divalent cations; the monomer–dimer–trimer equilibrium of HSC70 (*J58*); the single- and double-ringed heptameric *Thermoanaerobacter brockii* chaperonin 60 protein (Tbr-EL7) and its complexes with *E. coli* GroES and *T. brockii* TbrES (*J59*); human Hsp70 and *E. coli* DnaK proteins and their complexes with several unfolded substrate proteins (*J60*); DnaK complexed with carboxymethylated lactalbumin, RepA, and other proteins and the modulation of complex formation of these proteins by DnaJ in an ATP-dependent manner (*J61*, *J62*); and the ADP-dependent complexes formed between GroEL and GroES or spinach chloroplast cpn21 (*J63*).

A method for miniaturized large-zone SEC analysis of protein association equilibria was described by Nenortas and Beckett (*J64*). The method has the advantage of reducing the time required for collecting the large-zone elution profiles while requiring modest amounts of protein. The method was applied to determining the equilibrium constant for the dimer/tetramer association of human oxyhemoglobin A₀, with results that agreed

well with literature values, as did the derived values for the dimer and tetramer Stokes radii. Schiffer and Stevens (*J65*) reviewed the use of the small-zone SEC method for the analysis of binding stoichiometry and affinity, with specific reference to antibody-antigen interactions.

Liu et al. (*J66*) studied the interaction of a recombinant humanized anti-IgE monoclonal antibody (rhuMAb E25) with a monoclonal human IgE antibody, using SEC to characterize and isolate immune complexes for analysis by analytical centrifugation. Chimeric immunotoxin monoclonal antibodies were expressed in *E. coli* and purified, and their tendency to aggregate under various environmental conditions was studied (*J67*). Regions of the immunotoxin which stabilize the structure were determined by site-directed mutagenesis, combined with analysis of the aggregation state by SEC.

Oligomerization of proteins requires specific regions of contact between subunits. The analysis of the aggregation state of multisubunit proteins by SEC, and of mutant polypeptides which have deleted or substituted sequences, can yield information on the sites of subunit contact when such experiments are not complicated by the possibility of global conformational changes in the folded subunit. Kohn et al. (*J68*) studied a series of specifically substituted synthetic peptides, which were designed to form (dimeric) α -helical-coiled coils. SEC with and without LALLS was employed to determine the aggregation state of these peptides under varying environmental conditions. A number of studies used deletion mutagenesis to define sequences required for assembly of recombinantly expressed proteins. Analysis of avian sarcoma virus integrase revealed that deletions in both the catalytic core and C-terminal domains are required for dimerization and formation of biologically relevant higher oligomers (*J69*). Similarly, deletion mutagenesis and SEC analysis of dimerization capability was applied to the study of membrane GC-A guanylyl cyclase to define a 43-residue intracellular domain required for the formation of catalytically active dimeric enzyme (*J70*). Mende et al. (*J71*) conducted hydrodynamic characterization of G protein γ_2 subunit dimers and investigated the conditions under which deletion mutants could form complexes with the G protein β_1 and α subunits. Potter and colleagues (*J72*) used SEC to compare the ability of troponin I and a 57-residue N-terminal truncated mutant to form complexes with troponin C and T.

The bifunctional enzyme formiminotransferase cyclodeaminase is a tetramer of dimers. To determine the sites of catalytic activity, a series of N- and C-terminal deletion mutants were constructed by Murley and MacKenzie (*J73*). SEC analysis of the mutants showed that the isolated catalytic activities reside at either terminus and that the isolated domains self-associate to form homodimers. This finding implicates two distinct subunit interaction sites for the formation of the octameric complex. To investigate the role of the T1 intracellular N-terminal domain of the Shaker voltage-gated potassium channel, truncated recombinant protein domains were radiolabeled during *in vitro* translation, and the resulting products were analyzed by SEC and SDS-PAGE (*J74*). Chemical cross-linking of the two peaks seen in SEC demonstrated that the monomeric T1 domains formed a tetramer, without appreciable amounts of stable dimer or trimer forming.

Deletion polypeptides and isolated protein domains can also be obtained by specific proteolytic cleavage. Treatment of skeletal muscle actin with subtilisin permitted preparation of subactin, which remained folded but lacked a linking peptapeptide (*J75*).

SEC analysis was performed to demonstrate that the resulting protein retained its DNAase I associability. Specific proteolysis was also conducted to prepare domains of human coagulation factor IX (*J76*). SEC was applied for the hydrodynamic characterization of the isolated domains, to study the conformational changes in the domains induced by Ca^{2+} , and to investigate the stoichiometry of complexes formed on mixing the isolated domains.

Membrane protein conformation and structure analysis remains an area of great challenge. Gramicidin A is a highly hydrophobic, membrane-channel-forming peptide that has been extensively studied as a model system of intrinsic membrane proteins. Greathouse et al. (*J77*) studied the conformation and aggregation state of gramicidin A dispersed in diacetylphosphatidylcholines of varying chain lengths, finding a strong interdependence between gramicidin conformation and organization of micellar structures. The retinal heterotrimeric G-protein transducin is composed of a dissociable α subunit and a nondissociable $\beta\gamma$ heterodimer, which are modified with N-acyl and N-farnesyl lipids, respectively. To gain knowledge of the possible functional importance of these lipid modifications, the effects of the presence and absence of these lipid modifications on the association of α and $\beta\gamma$ were studied by SEC in the presence of a variety of surfactants, phospholipids, and cofactors (*J78*).

The greatest challenges to analyses of interconverting species by SEC are that (1) analytes passing through the SEC column suffer dilution in the mobile phase and (2) species which convert with kinetics faster than or comparable to the time scale of the chromatographic separation will continuously change their concentrations during the separation process. Although a variety of calculation procedures have been employed to reconstruct or model the band shape and area of eluting peaks in order to obtain quantitative information on equilibrium concentrations and the kinetics of interconversion, evaluation of such systems remains a challenging task. Several recent publications have analyzed kinetics of protein associations, in which species conversion is relatively slow, avoiding the need for detailed mathematical analysis of the SEC data.

The bacteriophage T4 replication helicase (gp41) was observed by Dong et al. (*J79*) to undergo a concentration-dependent dimerization in the absence of purine nucleoside triphosphates (PuTPs). The presence of PuTPs drove the assembled dimers into a hexameric assembly, which required ultracentrifugation for analysis. Cho and Cummings (*J80*) similarly analyzed the association equilibrium of native and mutant recombinant galectin 1 in the presence and absence of glycoprotein ligands. Recombinant proliferating cell nuclear antigen (*J81*) was also studied by SEC and its combination with chemical cross-linking. This protein undergoes concentration and Mg^{2+} -dependent oligomerization, exhibiting dimers and trimers with higher protein concentrations.

A detailed analysis of the kinetics of heterodimer formation was accomplished for the HIV-1 and HIV-2 reverse transcriptases by Divita et al. (*J82*). Yang et al. (*J83*) noted biphasic kinetics for the heat-induced dissociation of recombinant horse apoferritin.

Current thoughts on protein folding pathways recognize that a variety of schemes may apply to describe the transition of unfolded polypeptide to the native folded structure. In recent years, evidence has accumulated on the existence of folding intermediates, which may appear under certain environmental

conditions, the best known of these is the molten globule state, which is also variously known as the "compact intermediate" or "collapsed form". Other intermediate states are known to exist. A number of model schemes have been described in recent studies using SEC for the analysis of protein folding. Experimentally, protein folding patterns are frequently investigated by physicochemical analysis of equilibrium intermediates formed by varying the concentration of denaturing agents, such as urea or GuHCl, by altering the pH, or by elevating the temperature.

Studies by Uversky (J84) and Zerovnik et al. (J85) have employed SEC to detail the characteristics of two-state denaturant-dependent equilibrium unfolding (exemplified by myoglobin and lysozyme), as well as those which unfold via the molten globule state (e.g., bovine and human α -lactalbumins, bovine carbonic anhydrase B, β -lactamase from *Staphylococcus aureus*, and human stefins). At lower temperatures, several of these proteins also exhibit a state intermediate in size between the molten globule and the fully unfolded state of proteins, the so-called partly folded intermediate (J86). Analyses of these four-state unfolding patterns illustrate the high information content of SEC measurements and place such measurements in context with the information that can be obtained by near- and far-UV circular dichroic spectroscopy, the ANS fluorescence binding assay, and enzyme activity measurements. Importantly, SEC has the advantage of permitting measurements of individual folding intermediates, whereas spectroscopic measurements refer to average values for mixtures of folding intermediates.

To study the refolding of *E. coli* serine hydroxymethyltransferase, which was denatured with urea, Cai et al. (J87) used SEC to follow the rate of dimer formation from a structured monomer. Analytical-scale SEC proved useful for establishing solution conditions which would permit the formation of a stable folding intermediate of reduced lysozyme (J88). Unfolding of the dimeric trp aporepressor from *E. coli* was studied by Eftink et al. (J89). This study suggests a three-state equilibrium between the native dimer, a structured monomer, and a fully unfolded state. In contrast, no intermediate states were observed by SEC during the unfolding of porcine class π glutathione S-transferase (J90), which exhibited a direct transition from native dimer to unfolded monomers. Inouye et al. (J91) studied the complex case of heat and urea dissociation and unfolding of superoxide dismutase. The analysis of this enzyme is complicated by the concentration-dependent formation of active monomers and the urea- and temperature-induced disassociation of the active dimer into both active and inactive monomeric forms.

To investigate the role of polypeptide folding on the ability of melittin to self-tetramerize, a series of synthetic peptides substituted at position 7 (lysine) were studied by SEC at high and low ionic strength (J92). The formation of biologically active tetramer required that the amphipathic peptide was able to adopt a helical conformation. Nagai et al. (J93) studied the role of asparaginyl-linked oligosaccharides on the folding and assembly of soybean lectin tetramers.

Establishing appropriate conditions for the refolding of chaotrope-denatured recombinant proteins is a general problem of considerable importance. Many proteins expressed at high abundance in *E. coli* will be found in an insoluble form in the inclusion bodies. In general, these proteins require solubilization with guanidine or urea. An example of the use of SEC for monitoring the formation of correctly refolded protein from a

guanidine solution was illustrated for recombinant interleukin 4 (J94). Werner et al. (J95) suggest that on-column SEC protein refolding from solubilizing denaturants may be an effective general method. Several examples of proteins and complexes prepared by on-column refolding were presented.

The slow kinetics of conversion of native and denatured basic fibroblast growth factor under specific conditions permitted the SEC separation and quantitation of native and denatured species (J96, J97). This technique allowed detailed investigation of the formulation stability and permitted analysis of denatured degradation products.

MICROCOLUMN SEC

An interference-polarizing refractometer for SEC microcolumns of 0.2–0.5 mm i.d. was developed and evaluated by Alexandrov et al. (K1). The detection limit at a signal-to-noise ratio of 3 was 8×10^{-8} refractive index units. This group (K2) also considered problems related to microcolumn SEC in terms of retention time reproducibility.

PREPARATIVE SEC

Janson (L1) reviewed process-scale SEC for biopolymers in terms of separation principles, column packings, adsorption effects, mobile phase selection, sample throughput, and scale-up. Vemuri and Rhodes (L2) used large-scale process SEC to separate liposomes from free drugs (unencapsulated) in encapsulated drug preparations. Kurnik et al. (L3) evaluated three industrial-scale methods for buffer exchange of proteins: SEC, tangential flow filtration, and countercurrent dialysis. Preparative-scale SEC was used by Tyler and Ratner (L4) to fractionate a chemically heterogeneous poly(ether urethane) sample. Fractions were then characterized and tested for susceptibility to oxidative degradation by exposure to hydrogen peroxide. Other examples of preparative SEC include the fractionation of humic acids (L5) and CdS sols with particle diameters of 2–8 nm (L6).

COUPLED COLUMNS/COLUMN SWITCHING

Janco et al. (M1) described an eluent switching approach in which an adsorption column is coupled to an SEC column. In the example given, polystyrene and PMMA are injected using toluene as the mobile phase. PMMA is retained on the adsorption column (silica), and polystyrene is eluted in the SEC column (cross-linked polystyrene). After the eluent is switched from toluene to THF, PMMA is desorbed and separated by size exclusion on the second column. A similar approach was reported by Nerin and co-workers (M2); however, the SEC column preceded the adsorption column. After SEC of the polymer was complete, low molecular weight solutes, i.e., antioxidants and UV stabilizers, were diverted into the second column (silica) and separated via normal-phase adsorption.

Dawkins (M3) discussed copolymer characterization by SEC using first concentration detectors with low-angle laser light-scattering detection and second, concentration detection with on-line transfer to an HPLC column. Examples given were polystyrene, poly(butyl methacrylate), and styrene–butyl methacrylate copolymers. Kilz et al. (M4) described an automated HPLC-SEC system for determining the chemical composition distribution of complex copolymers. Gradient HPLC was used to separate a multicomponent star block copolymer mixture followed by SEC. In a second example, the HPLC-SEC system was used to

characterize telomeric aliphatic polyesters in terms of end-group differences.

Moore and Jorgenson (*M5*) used SEC in the first dimension to separate proteins, followed by reversed-phase separation, and then into a capillary zone electrophoresis system. Cortes et al. (*M6*) used a multidimensional approach for the analysis of polymer additives. In their system, a micro-SEC column was coupled to a capillary GC column or to an HPLC system, depending on the volatility of the solutes.

FIELD-FLOW FRACTIONATION

Reviews on field-flow fractionation (FFF), with emphasis on macromolecular separation, are given in refs N1–N4. Sisson and Giddings (*N5*) studied the effects of solvent composition on thermal FFF retention of polystyrene. van Asten et al. (*N6*) reported on temperature dependence of solvent viscosity, solvent thermal conductivity, and Soret coefficient in thermal FFF of polystyrene in THF and ethylbenzene. This group (*N7*) investigated separation speed in thermal FFF. Off-line coupling of SEC with thermal FFF was used by van Asten et al. (*N8*) to cross-fractionate copolymers and polymer blends. Since thermal diffusion is strongly affected by the chemical nature of the polymer, the chemical composition can be studied as a function of molecular weight. Examples used were polystyrene blended with polybutadiene and polytetrahydrofuran, and butadiene- and styrene-methyl methacrylate copolymers. The validity of universal calibration in thermal FFF is discussed in ref E16.

Schimpf et al. (*N9*) reported that the thermal diffusion coefficients for random and block isoprene-styrene copolymers in THF and cyclohexane were a linear function of copolymer composition. Also studied was thermal diffusion in liquid mixtures and its effect on polymer retention (*N10*). Schimpf (*N11*) presented a method for obtaining the molecular weight and composition of copolymers using thermal FFF and viscometry.

Thermal FFF was used to determine the molecular weight and molecular weight distribution of natural rubber (*N12*). As compared to SEC, thermal FFF yielded more accurate results owing to its higher resolution for the ultrahigh molecular weight species. (Also see ref C5, which compares the resolution of thermal FFF, SEC, and hydrodynamic chromatography). Lee and Kwon (*N13*) characterized ultrahigh molecular weight poly(methyl methacrylate) using thermal FFF with a light-scattering detector. Antonietti et al. (*N14*) coupled thermal FFF to an on-line MALLS detector for determining the molecular weight distribution of polystyrene, sulfonated polystyrene, microgels, and block copolymer micelles. The thermal diffusion and molecular weight of poly(ethylene-co-vinyl acetate) samples were measured by thermal FFF (*N15*, *N16*).

On-line coupling of flow FFF and MALLS detector was accomplished by Roessner and Kulicke (*N17*) for polystyrene particles and dextran macromolecules. Hollow-fiber flow FFF was used by Wijnhoven et al. (*N18*) for the characterization and separation of sodium poly(styrene sulfonate) samples. Hanselmann and colleagues (*N19*) characterized starches with sedimentation FFF and an on-line MALLS detector.

SELECTED APPLICATIONS

Synthetic Polymers. Comprehensive SEC reviews have been published on acrylamide homopolymers and copolymers (*O1*), copolymers (*O2*), polyamides, polyesters, and fluoropoly-

mers (*O3*), poly(vinyl acetate) (*O4*), poly(vinyl alcohol) (*O5*), natural and synthetic rubbers (*O6*), and vinylpyrrolidone homopolymers and copolymers (*O7*).

Reports have been written on the SEC of alkyd resins (*O8*), ethyl(trimethylsilyl)ethynylthiophene homopolymers (*O9*), fluorocarbon elastomers (*O10*), fluoropoly(oxyalkylenes) (*O11*), perylene dyes (*O12*), phenol-formaldehyde resins (*O13*), polyacrylamide (*O14*), poly(acrylic acid) (*O15*), polyanilines (*O16*), poly(ethers) (*O17–O19*), poly(organometallosiloxanes) (*O20*), polypropylene (*O21*), propellants (*O22*), and rubbers (*O23–O25*).

Asphalts, Bitumens, Fossil Fuels, and Related Products.

An in-depth review was written on SEC of asphalts (*O26*). Asphalt performance and properties as related to SEC analysis can be found in refs O27–O31. SEC has been used for the analysis of coal tar pitch (*O32*), hard coal extracts (*O33*), brown coal-derived preasphaltenes (*O34*), asphaltene fractions (*O35*, *O36*), and metalloporphyrins from heavy crude oil (*O37*).

Polysaccharides and Cellulosics. Detailed reviews were published on the SEC of starch (*O38*), cellulose and cellulose derivatives (*O39*), and carbohydrates and glycoconjugates (*O40*). SEC has been reported for cellulose (*O41*, *O42*), cellulose ethers (*O41*), cellulose derivatives (*O42*), cellulose nitrate (*O43*), hydroxyethyl cellulose (*O44*), chitosan (*O45*), cotton (*O46*), amylose and amylose pectin (*O47*), starches (*O48*), hyaluronic acid (*O49*, *O50*), oligosaccharides (*O51*), water-insoluble β -D-glucan (*O52*), glycoproteins (*O53*), and dietary fiber (*O54*).

Lignins and Tannins. A general review of the SEC of lignin derivatives was given by Himmel et al. (*O55*). Studies involving SEC of lignin can be found in refs O56–O60. Hydrolizable tannins, which were derivatized, were characterized by SEC using THF as the mobile phase (*O61*). DMF was used for the SEC analysis of procyanidin oligomers and polymers in their free phenolic form (*O62*).

Natural Oils, Fats, and Food Products. Firestone (*O63*) reported on an IUPAC collaborative study involving the SEC analysis of polymerized triglycerides in fats and oils. A similar interlaboratory study undertaken in the Netherlands was described by Baljaars et al. (*O64*). SEC was used to analyze the quality of vegetable oils during industrial refinement (*O65*), to study the autoxidation of triacylglycerol (*O66*), and to analyze non-urea-adduct fraction in used soybean oil (*O67*). Fillieres et al. (*O68*) used SEC to monitor the transesterification of rapeseed oil with ethanol. Monoglycerides obtained from the glycerolysis of fatty oils were reported by Pham et al. (*O69*). Marquez-Ruiz et al. (*O70*) used SEC to characterize mixtures of sucrose polyesters-triacylglycerols. Total chlorogenic acid, trigonelline, and caffeine were determined in green coffee by SEC (*O71*).

Inorganic Compounds and Particles. Kirkland (*O72*) reviewed methods for characterizing silica sols, including SEC, HDC, and FFF approaches. Cadmium sulfide and zinc sulfide colloidal semiconductor particles of 2–20 nm were analyzed by SEC using silica packings with pore sizes of 30–100 nm (*O73*). Fetuin-coated poly(lactic acid) particles of 50–200 nm were separated with Sepharose CL-2B and Sephacryl S1000 (*O74*). Bio-Gel P2 and Fractogel HW-40 were used to separate metal chelates (*O75*).

Membrane Characterization. Bodzek and Korus (*O76*, *O77*) used SEC to characterize ultrafiltration membranes. This was accomplished by determining the molecular weight distribution of the feed and permeate streams.

Sample Cleanup/Pretreatment. SEC is a useful method for the cleanup of environmental samples, followed usually by GC or HPLC analysis of fractions. Examples include the determination of pesticides and/or polychlorinated biphenyls in soils and sludges (O78–O81), animal fats (O82–O85), crops (O86, O87), feeds (O88), and other environmental samples (O89); polycyclic aromatics in lanolin (O90); organic and inorganic phosphates in water and sediments (O91); and metalloporphyrins in crude oils (O92).

Blomberg et al. (O93) described an automated sample cleanup system based on the coupling of SEC to a GC for the determination of additives in polymer matrixes. As a precolumn cleanup for the HPLC of a drug in plasma, Stopher and Gage (O94) developed an automated system for removing protein and lipids by means of a Sephadex G25 column coupled to the HPLC system.

Biopolymers. Analytical SEC biopolymer applications are now dominated nearly completely by the use of small-particle (<20 μm diameter) agarose- and silica-based hydrophilic packing materials. Research applications favor the use of the less mechanically rigid agarose materials, whereas high-throughput applications favor silica-based column packing materials. Table 1 presents selected examples of SEC separations and analytical methods of biochemical interest. These are presented both to illustrate the broad range of sample types for which SEC has been successfully applied and to direct the reader to separation conditions which may be related to a problem of interest.

Proteins, Peptides, and Conjugates. Protein chemists continue to make heavy use of SEC for preparative separations, as well as for an analytical tool to monitor protein purification regimes and to study formulation stability. A surprising number of proteins show concentration-dependent oligomerization (see also section J on Physicochemical Studies).

Methods for the analysis of immunoglobulin purity were described by Page et al. (O95). A method for the quantitation of immunoglobulins and other whey proteins in bovine colostrum was presented (O96). Muddukrishna and colleagues (O97, O98) describe improved methods for the analysis of technetium-radiolabeled monoclonal antibodies (MAbs) and for the quantitation of free sulfhydryl groups in MAbs. Costa et al. (O99) developed an SEC method for the quality control of a recombinant hepatitis B antigen for use in vaccines.

SEC analysis of pharmaceutical and veterinary hormones and proteins continues to be widely used for research, quality control, and formulation stability studies. Selected examples of recent interest include fibroblast growth factor (J96, J97), insulin and dextran conjugates of insulin (O100, O101), human factor IX (O102), bovine somatotropin (O103, O104), and recombinant human growth hormone (O105–O109).

The application of SEC to the detection and quantitation of ligand binding to protein receptors remains in broad use. Asami et al. (O110) used radiolabeled ouabain with SEC to quantify the amounts of the solubilized Na^+/K^+ pump in purified protein preparations and tissue homogenates. The levels of free and protein-bound cyclosporin and its metabolites were studied in human blood (O111). Owen et al. (O112) used SEC coupled with ICPMS to determine aluminum, copper, and zinc in their bound forms in the soluble fraction of intestinal digesta. A spun-column SEC method was optimized, and described in detail (O113), as a general tool for the study of protein–ligand interactions.

Analytical SEC methods were described for a variety of food proteins, including the primary protein components of whey (O114); hydrolysates of casein (O115); albumins (O116), glutenins and gliadins (O117), and other protein components of wheat (O118, O119); and the proteins of soybeans (O120, O121).

SEC combined with immunochemical detection can be very useful for the investigation of polypeptide processing, as demonstrated in the comparison of chromogranin A processing in several human cell lines by Brandt et al. (O122). Nylander et al. (O123) used SEC coupled with ESI-MS to investigate the processing of prodynorphin in rat brain. Stroemqvist (O124) used SEC in combination with reversed-phase and capillary electrophoretic separations to characterize the peptides resulting from the tryptic digestion of recombinant superoxide dismutase. Libera et al. (O125) used SEC separation of the components of muscle cell culture medium to characterize and partially purify a component which inhibited tumor cell proliferation in culture. Plasma lipoproteins were resolved by SEC for analysis of patient lipoprotein phenotype (O126).

Nucleic Acids. Kato reviewed the use of SEC for the isolation and analysis of plasmid DNAs, DNA restriction fragments, and various RNA species (O127). The use of SEC for the final polishing step in the preparation of high-purity plasmid DNAs was described (O128). Akane et al. (O129) used SEC for the purification of DNA fragments as templates for polymerase chain reaction amplification from degraded forensic specimens.

Others. An SEC method for fractionation of oligosaccharides was presented by Kobata (O130). SEC analysis of pectin digestion products by crude commercial enzymes (O131) and purified recombinant enzyme (O132) was conducted to characterize the activities of the enzymes and sites of attack on pectins. Song et al. (O133) size-fractionated crude cellulase of *Trichoderma viride* and studied the enzymatic activity of the fractions. The activity and enzymatic mechanism of testicular hyaluronidase was characterized by analysis of hyaluronan hydrolysates (O134).

Dobarganes and Marquez-Ruiz (O135) reviewed the use of SEC for the analysis of lipids, including triglycerides, oxidized lipids, lipoproteins, vesicles, and micelles. Hopia (O136) studied the high molecular weight oxidation products of soybean, rapeseed, and sunflower oils.

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