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Infrared Spectroscopy

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Review Contents

This review covers the published literature for the period late 1991 to October 1993 on aspects of infrared spectroscopy that are relevant to chemical analysis. Our review has a strong bias toward papers published in English, or in certain aspects of IR spectroscopy that are of particular interest to one or more of the co-authors. In addition, a few selected references to FT-Raman spectroscopy are included for reasons given below.

OVERVIEW OF ANALYTICAL INFRARED SPECTROSCOPY

Infrared radiation is usually defined as that electromagnetic radiation whose frequency is between ~14 300 and 20 cm^{-1} (~0.7 and 500 μm). Within this region of the electromagnetic spectrum, chemical compounds absorb IR radiation providing there is a dipole moment change during a normal molecular vibration, molecular rotation, molecular rotation/vibration, or a lattice mode or from combination, difference, and overtones of the normal molecular vibrations. The frequencies and intensities of the IR bands exhibited by a chemical compound uniquely characterize the material, and its IR spectrum can be used to identify and quantify the particular substance in an unknown sample.

Different classes of chemical compounds contain chemical groups which absorb IR radiation at essentially identical frequency(ies) and have essentially the same band intensity(ies) within each class of compound, and these bands are termed "group frequencies". Group frequencies are predictable and allow the analyst to elucidate and identify molecular structures without available IR standard spectra for comparison. In addition, IR spectra can be recorded rapidly of materials in the solid, liquid, solution, and vapor phases over a wide range of temperature. Such studies aid in elucidating the molecular structure of materials in different physical phases.

Today modern IR instrumentation allows spectra to be recorded of samples available in only low picogram quantities. No other technique allows examination and identification of materials under such a wide variety of physical conditions, and it is this versatility that has allowed IR spectroscopy to develop into the "work horse" of analytical science.

One should be aware of the fact that Raman spectroscopy is a technique complementary to IR spectroscopy. In cases where a chemical compound has a center of symmetry, certain normal vibrations will only be active in the Raman and certain normal vibrations will only be active in the IR. Thus, one needs both techniques to record the complete vibrational spectrum of many chemical compounds. Moreover, bands which are strong in the Raman are usually weak in the IR and vice versa in cases where the normal modes are allowed in

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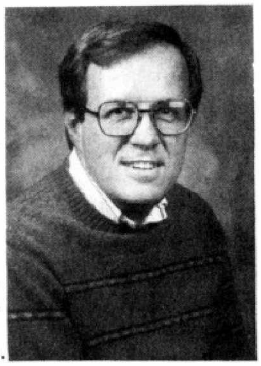

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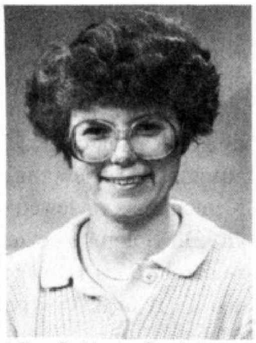


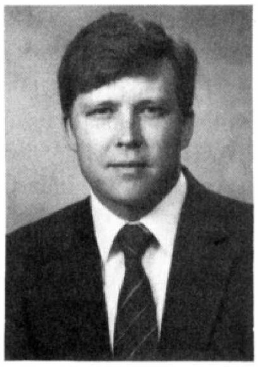
Marianne L. McKelvy is a Research Leader in the Materials Characterization Group of the Analytical Sciences Laboratory, The Dow Chemical Co., U.S.A. She received the B.S. degree from the University of Detroit, Detroit, MI (1979) and the M.S. degree (1982) and the Ph.D. (1985) from Polytechnic University, Brooklyn, NY. Subsequently, she joined the Analytical Sciences Laboratory, where she is involved in solving polymer problems using infrared spectroscopy. Her research interests involve the characterization of polymers using vibrational spectroscopy and infrared microspectroscopy. She is a member of the Society for Applied Spectroscopy and the Coblentz Society.

both vibrational techniques. With the recent development of Fourier transform Raman (FT-Raman), it is now possible to rapidly record Raman spectra of most materials by using a FT-IR system equipped with the FT-Raman accessory. FT-Raman is commercially available from most manufacturers,and we predict that in the future both IR and Raman spectra of materials will be recorded on a routine basis for the elucidation and identification of molecular structure. Therefore, we have included a few selected references to FT-Raman spectroscopy for your convenience.

(A) BOOKS

Shagidullin et al. published an atlas of IR spectra of organophosphorus compounds (*A1*). Suzuki et al. published an IR and Raman literature data base for the period June 1990–May 1992 (*A2*). Harrington and Katzir edited a book on fiber optics (*A3*). Jajadev edited a book on IR sensors, detectors, electronics, and signal processing (*A4*). Durig edited the 20th volume of *Vibrational Spectra and Structure* (*A5*).

Richard A. Nyquist is a Research Scientist the Analytical Sciences Laboratory, The Dow Chemical Co., U.S.A. He received his B.A. degree in chemistry from Augustana College, Rock Island, IL, and his M.S. degree from Oklahoma State University. He joined The Dow Chemical Co. in 1953, and his career has been mainly in the field of vibrational spectroscopy. He utilizes IR and Raman spectroscopy for solving chemical problems, for the elucidation of molecular structure, and for qualitative and quantitative analyses. Nyquist is the author or coauthor of over 150 scientific articles including books, chapters in books, and patents. He is a member of the American Chemical Society and the Society for Applied Spectroscopy. In 1985 he received the Williams–Wright Award for his contributions to industrial infrared spectroscopy from the Coblentz Society. Dick is an honorary member of the Coblentz Society, and in 1989 he was a national tour speaker for the Society of Applied Spectroscopy. In 1993 he was honored with the ANACHEM Award from the Association of Analytical Chemists for his contributions to analytical chemistry.



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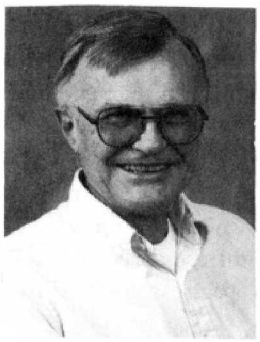

Richard R. Papenfuss is a Research Associate with the Organic Group, the Analytical Sciences Laboratory, The Dow Chemical Co., U.S.A. He received his B.S. degree from Wisconsin State College at LaCross. He joined Dow in 1963, and has worked in the Inorganic, Polymer and Agricultural Product Groups. For the last 14 years, he has concentrated in applying both qualitative and quantitative infrared spectroscopy to polymeric systems. In addition to continuing his work in infrared, he is currently applying gas chromatography/infrared spectroscopy to polymeric and other related materials.

Lori Yurga is a Project Leader in the Administration Group of the Analytical Sciences Laboratory, The Dow Chemical Co., U.S.A. She received a B.S. degree in chemistry and biology from Central Michigan University and joined The Dow Chemical Co. in 1983. She worked for two years in methods development involving separation science in the Organic Analysis Group prior to her assignment to the Instrumental Methods Group, where her expertise was concentrated in problem solving using infrared and Raman spectroscopy. Her research interests are in the development and application of hyphenated techniques combining chromatography and infrared spectroscopy. She currently serves as the Quality Performance Coordinator for the Analytical Science Laboratory.

(B) REVIEWS

A review of IR spectrometry covering the period late 1989 to late 1991 has been published (*B1*). Thomas has reviewed the early history of spectroscopy (*B2*).

Miller has identified the origins and developers of several widely used IR techniques (*B3*). Domingo and Escribano have reviewed the IR and Raman new experimental techniques, rovibrational theory, and new trends in applications (*B4*).

Koenig reviewed the field of FT-IR spectroscopy of polymers with emphasis on spectral scattering, reflection, spectral distortion, spectral subtraction, and curve fitting (*B5*). Connes has written the history of the early development of FT-IR at the Laboratoire Aime Cotton under P. Jacquinot's direction over the years 1954–1963 (*B6*). Miroshnikov reviewed the history of the development of IR in the Soviet Union (*B7*).

Ferraro et al. reviewed the field of commercial landmark IR and Raman instrumentation (*B8*). Baumeister and Wilkins reviewed the computer-enhanced fields of GC/IR, GC/IR/MS, etc. (*B9*). Davidson and Jenkins reviewed the field of SFC/FT-IR (*B10*), Taylor and Calvey reviewed the field of flow cell SFC/FT-IR (*B11*), and Bartle et al. reviewed practical applications of SFC/FT-IR (*B12*).

Fringeli reviewed the field of in situ ATR/IR experiments with emphasis for its application in drug design and delivery (*B13*). Grasselli reviewed the applications of IR and Raman spectroscopy in industrial problem solving (*B14*). Fringeli reviewed the field of in situ ATR/IR membrane spectroscopy (*B15*). Coates reviewed industrial applications of internal reflection spectroscopy (*B16*), and Yarwood reviewed applications of ATR/IR for surface analysis (*B17*).

Palmer reviewed step-scan FT-IR as a versatile tool for time and phase-resolved vibration spectroscopy (*B18*). Hollins reviewed the affects of surface defects on the IR spectra of adsorbed species (*B19*). Reffner et al. reviewed the area of chemical microscopy of surfaces using grazing angle and ATR/FT-IR microscopy (*B20*).

Burrows reviewed ATR/IR for the chemical analysis of semiconductor surfaces, at interfaces, and in thin films (*B21*). Roberts reviewed the techniques of surface analysis using diffuse reflectance FT-IR, ATR/FT-IR, and photoacoustic spectroscopy (*B22*).

Cook reviewed the techniques and applications of IR and Raman microscopy in the analysis of small samples and small areas (*B23*). Katon and Sommer reviewed routine sampling methods extended to the microscopic domain (*B24*). Kirkbride reviewed the application of IR microscopy for the analysis of single fibers (*B25*).

Winnewisser reviewed the strategies for the exploitation of intensity information obtained from high-resolution FT-IR spectra (*B26*). Steele reviewed the intensity distribution in IR and Raman vibrational bands (*B27*). Tennyson and Miller reviewed the field of calculating the molecular spectra using vibration/rotation data for small molecules (*B28*), and Graner reviewed the determination of accurate molecular structure by application of vibration/rotation spectroscopy (*B29*). Warrington reviewed the area of evolved gas analysis using FT-IR and mass spectroscopy (*B30*). Howard and Brown reviewed high-resolution IR spectroscopy (*B31*). Melen and Herman reviewed the vibrational data for $H_xN_yO_z$ compounds (*B32*).

Milicev reviewed the vibrational spectroscopy of inorganic coordination compounds of fluorine (*B33*). Swalen reviewed the past, present, and future techniques for the analysis of Langmuir–Blodgett films, which include IR and Raman spectroscopy (*B34*). Hirota reviewed the application of IR and microwave in the analysis of organic and inorganic free radicals and ions (*B35*). Brown et al. reviewed IR and NMR applications for the analysis of homogeneous catalysis (*B36*). Lercher reviewed the kinetics of catalyzed processes by application of FT-IR spectroscopy (*B37*).

Mantsch reviewed bioanalytical applications of FT-IR (*B38*). Filippov reviewed the application of IR in the analysis of pectic substances (*B39*). Cooper reviewed the role of IR spectroscopy in the analysis of ceramics (*B40*).

Jackson and Mantsch reviewed the application of FT-IR in the analysis of biological membranes (*B41*). Nafie reviewed instrumental techniques for the measurement of vibrational optical activity (*B42*).

Birch and Yarwood reviewed far-IR and microwave, including the theoretical background, spectroscopic considerations, experimental methods, and interpretation of spectral data (*B43*). Fuller and Ottenroth reviewed the theory and application of diffuse reflection IR spectroscopy (*B44*). Julien reviewed far-IR instrumentation, far-IR experimental techniques (*B45*). Mills et al. reviewed applications of IR in drug analysis (*B46*). Sabot et al. reviewed the identification of drugs and metabolites in urinary calculi by application of IR (*B47*). Doyle reviewed the area of continuous monitoring of organic pollutants in water by sparging and the application of IR spectroscopy (*B48*). Van de Voort reviewed the application of FT-IR in the analysis of food (*B49*).

Hansen reviewed on-line monitoring of polymeric processes by application of IR (*B50*). Hurst and Martin reviewed the automation of new analytical technologies including IR (*B51*). Kemeny has reviewed the use of near-IR in process analysis (*B52*). Solomon reviewed the use of on-line FT-IR in coal research (*B53*). Doyle reviewed the principles and applications of FT-IR in process analysis (*B54*).

Noda et al. reviewed two-dimensional correlation IR spectroscopy (*B55*). Stoutland et al. reviewed ultrafast IR spectroscopy (*B56*). Sloan and Kruus reviewed time-resolved FT-IR spectroscopy (*B57*).

Dundas has reviewed the literature on the detection of hydrocarbons using IR (*B58*, *B59*). Chabal has reviewed the literature on vibrational spectroscopy of adsorbates on silicon surfaces (*B60*). Smith et al. have reviewed the literature for the analysis of synthetic polymers and rubbers using various techniques including IR and Raman spectroscopy (*B61*). Jinno has reviewed the application of FT-IR coupled with micro-column liquid chromatography for polymer characterization (*B62*).

Comyn reviewed surface analysis and adhesive bonding techniques including surface IR spectroscopy (*B63*). Kalasinsky and Kalasinsky reviewed the field of HPLC/FT-IR (*B64*). Kalasinsky et al. compared the application of IR and MS in drug analysis (*B65*).

Urban reviewed surface and interface vibrational spectroscopy as related to studying adhesion (*B66*). Tascon et al. reviewed IR spectroscopy and temperature-programmed desorption of perovskite surfaces by application of IR (*B67*).

(C) FAR-IR TECHNIQUES AND APPLICATIONS

Far-IR spectroscopy, because of its sensitivity to three-dimensional order, continues to be utilized primarily as a tool to gain information regarding physical structure of solid materials and geometry of molecules and macromolecules.

Application to Molecules and Macromolecules. Preliminary measurements on substrate-supported films of five oligonucleotides were reported for various conditions of temperature and humidity by Powell et al. (*C1*). Tsunashima et al. studied the zinc(II) salts of ethylene–methacrylic acid copolymer with 1,3-bis(aminomethyl)cyclohexane using far-IR spectroscopy (*C2*). The structure and transition of the ionic clusters are discussed. An investigation of the proton transfer with substituted phenol and *N*-mono- and *N,N*-dioxides as a function of the pK_a of the phenols was conducted by Keil et al. using far-IR spectroscopy (*C3*). Raman and far-IR spectra of 2,3-dichloropropene were measured, vibrational assignments made, and ab initio calculations performed by Durig et al. (*C4*) in order to estimate conformational stability and barriers to internal rotation in the molecule. Buchner and Yarwood reported their studies of the molecular dynamics and interactions in *N*-methylformamide using far-IR spectroscopy (*C5*). Rivera-Gaines et al. studied the far-IR spectra of cyclohexene and its isotopomers in order to evaluate the potential energy surface and the energy required for conformational changes (*C6*). Far-IR spectroscopy was employed by Zoidis et al. in order to study the interaction-induced absorption spectra of carbon disulfide/benzene liquid mixtures (*C7*). The intermolecular stretching vibrations of complexes between several Schiff bases and phenols were studied by Migchels and Zeegers-Huyskens (*C8*). Sciesinska et al. studied the far-IR spectroscopy of solid cyclohexane and deuterated analog for phases I and II at various temperatures (*C9*). Low-frequency spectra of Langmuir–Blodgett films of chlorophyll *a*, chlorophyll *b*, pheophytin *a*, and their adducts with water and dioxane were studied using far-IR measurements (*C10*).

Application to Solid-State Materials Characterization. Rotter and co-workers described the use of far-IR reflectivity measurements to probe the low-energy response of rubidium-doped fullerene. These measurements enabled the authors to derive a value for the characteristic energy scale associated with reflectivity and estimate the gap energy (*C11*). Lattice phonon modes in solid C60 fullerene were studied by Huant et al. using far-IR (*C12*). Far-IR reflectance spectroscopy was utilized by Fukasawa and co-workers (*C13*) in order to study heavily doped p-gallium arsenide at various hole concentrations. An investigation of the far-IR reflectance spectra of the nonlinear crystal, potassium pentaborate over the temperature range 90–300 K was performed by Miniewski et al. (*C14*). A study of the IR-active long-wavelength phonons of the layered semiconductor indium selenide was reported by Julien and Eddrief (*C15*). Julien et al. also reported on the far-IR reflectance of layer structured III–VI compounds (*C16*). Far-IR reflectance spectra of zinc selenide/zinc telluride strained-layer superlattices were measured on the structures grown by molecular beam epitaxy and reported by Cui and co-workers (*C17*). The low-frequency phonon modes of the superconductor yttrium barium copper oxide were measured and found to agree well with recent lattice dynamical calculations in a study by Litvinchuk et al. (*C18*). Gajic and co-workers measured the phonon softening in the far-IR spectra of yttrium barium copper nickel oxide (*C19*). The far-IR conductivity and reflectivity of charge density wave materials and the oxygen isotope effect in high-T_c superconductors was discussed by Creager (*C20*).

The pure rotational spectrum of hydrogen chloride in dense argon was analyzed by Medina and co-workers (*C21*) using two different stochastic bath descriptions. Far-IR Zeeman spectroscopy of shallow carbon and zinc acceptors in gallium arsenide was employed by Atzmueller and co-workers in order to understand the role of these impurities in the electronic and optical characteristics of this material (*C22*). Investigations of low-energy excitations in dilute magnetic semiconductors in high magnetic fields were conducted by Hausenblas et al. using fast-scan far-IR spectroscopy (*C23*).

An overview of the spectroscopy of the atmosphere using far-IR emission spectroscopy was presented by Russell (*C24*). The far-IR spectrum of carbon suboxide was recorded at 0.003 cm^{-1} and analyzed by Vander Auwera and co-workers (*C25*) in order to obtain improved constants and to calculate those for the higher quanta.

Barth and Keilmann reported the development of a far-IR rotating analyzer ellipsometer which uses a step-tunable, optically pumped gas laser as its light source (*C26*, *C27*). Specular and diffuse reflectance measurements of rough solid aluminum surfaces were measured in the far-IR by Smith (*C28*) for evaluation as "perfectly" diffuse and "perfectly" reflecting samples for calibration of reflectance measurements.

(D) NEAR-IR TECHNIQUES AND APPLICATIONS

The ease with which vibrational spectroscopic measurements can be performed remotely via silica core optical fibers has greatly expanded the use of near-IR spectroscopy in the last several years. Many near-IR fiber-optic-based intruments and probes are available for routine laboratory or process analysis in this spectral region. Unfortunately, the interpretation of the spectra is not as straightforward as the mid-infrared, and thus, the analysis relies heavily on the development of appropriate mathematical modeling especially for the analysis of complex matrices. The International Conference of Near Infrared Spectroscopy was held in 1991, and the conference proceedings were published in 1992 (e.g., ref D2). Many of the reports referenced below as well as others were published in that book, and it is an excellent review of the recent efforts in the area of near-IR spectroscopic applications.

Instrumentation. A novel approach to understanding vibrational features in the combination and overtone region was developed and presented by Barton et al. (*D1*). The analysis utilizes a two-dimensional statistical correlation of mid- and near-IR spectra in order to determine the most probable source of the near-IR signals. The development and characteristics of a high-speed, dual-beam acoustooptic tunable filter (AOTF) spectrometer for process analysis in the near-IR was reported by Kemeny (*D2*). A method of correcting near-IR emission spectra for variations in spectrometer response with wavelength was described by Petty et al. (*D3*). Calibration transfer and measurement stability of near-IR spectrometers was discussed by Wang and Kowalski especially with regard to gasoline samples (*D4*). The development of another design of an AOTF-based near-IR spectrometer was discussed by Huehne and co-workers (*D5*). The development of three small, rugged spectrometers for near-IR analysis was presented by Hyvarinen et al. (*D6*). Stark and Luchter reviewed the advantages and disadvantages of diode-array and optically encoded spectrometers (*D7*).

Biochemical Applications . The applicability of near-IR spectroscopic analyses to the field of clinical chemistry has received some attention (*D8*) in the last several years: in skin analysis (*D9*), analysis of urinary calculi (*D10*), and perinatal monitoring of cerebral oxidative metabolism (*D11*). Applications of near-IR analysis in the pharmaceutical industry have also been noted: in antibiotic production (*D12*), the assay of cephalosporin derivatives (*D13*), and identification of streptomycin (*D14*). Fast identifications of drugs for forensic science measurements was described by Kohn and Jeger using a fiber-optic probe and FT-near-IR spectrometer (*D15*). The use of near-IR flash absorption was evaluated as a tool for studying photosynthesis by Mathis and Thibodeau (*D16*). Bioconversion in a fermentation broth was evaluated by Varadi et al. (*D17*) using near-IR spectroscopy. The difficulty in assigning vibrational bands in the near-IR spectra of amino acids, di- and tripeptides, and proteins was discussed by Holly and co-workers (*D18*). Grant et al. invesigated the chemistry of polymer-associated water changes during glycosaminoglycan/polypeptide interaction (*D19*). Ozaki et al. reported the nondestructive and noninvasive monitoring of deoxyhemoglobin in the vein using fiber-optic near-IR reflectance spectroscopy (*D20*).

Chemicals and Polymers. Near-IR spectroscopy continues to be widely used in the production of chemicals and polymers. Martens and co-workers described a system which was developed for the process control of a steam cracker (*D21*). The dip add-on of resorcinol–formaldehyde latex adhesives for textile reinforcements and rubber components was analyzed using near-IR spectroscopy and reported by Knight (*D22*). A methodology for the end-point determination in poly(oxyethylene) esterification by near-IR spectroscopy was described by Mockel and Thomas (*D23*). Spatafore and McDermott compared the results of a near-IR reflectance method to other laboratory methods for the determination of primary and secondary antioxidants and light stabilizers in polyolefins (*D24*). The determination of cloud point for waxy crudes was shown to be viable using fiber-optic near-IR analyses by Alex and co-workers (*D25*). Zhu and Hieftje reported on analyses using near-IR spectroscopy for the determination of polymer composition and physical properties (*D26*). Brimmer et al. described the use of near-IR spectroscopy for the real-time monitoring of the polymerization reaction of polyurethane (*D27*). Bickel investigated the use of near-IR spectroscopy on line for the measurement of hydroxyl value in the production of polyols (*D28*). The design and implementation of a short-wave near-IR (700–1100 nm) spectrometer was presented by Lysaght for analysis of components in aviation fuel (*D29*). De Wit and Dugger described the use of near-IR spectroscopy for the analysis of cellulose esters (*D30*). The qualitative identification of fibers was demonstrated by Howell and Davis using near-IR spectroscopy (*D31*). Structural properties of nylon 66 fibers were examined by Rodgers and Lee (*D32*) by near-IR spectroscopy as well as X-ray crystallography in order to understand the effect of temperature changes in the sample. A patent disclosure was allowed for the apparatus and process of using near-IR spectroscopy for the determination of components in diesel fuel by Maggard (*D33*). The use of near-IR spectroscopic detection in thin-layer chromatography was demonstrated for the identification of components in pharmaceuticals by Ciurczak and co-workers (*D34*).

Food and Agricultural Analyses. Near-IR spectroscopy continues to be a widely used analytical tool for the characterization of various food products: sugar measurements (*D35–37*), grain and feed analysis (*D38–40*), protein analysis (*D41*), chromic oxide in feeds (*D42*), irradiation effects in paprika powder (*D43*), and fatty acids in oils (*D44*). Mid-IR spectroscopy was compared to near-IR spectroscopy for the analysis of lignocellulose, and near-IR was shown to be superior when all aspects of the analysis were considered in a study by Burns and Schultz (*D45*).

Chemometrics. While the review of chemometrics is beyond the scope of this review, mathematical postprocessing of near-IR spectra of extremely complicated samples such as grains, food, or fuels is crucial for the analysis. As such we provide a listing of some recent articles dealing with the application of chemometrics to near-IR spectroscopy (*D46–50*).

(E) IN SITU

Thin Films and Surfaces. An in situ cylindrical internal reflection FT-IR investigation of the effect of aromatic ring substituents on chemisorption onto titania ceramic membranes was conducted by Tunesi and Anderson (*E1*). Nguyen et al. reported on a technique for the in situ measurement of water at the coating/metal interface (*E2*). Blayo and co-workers described the design and operation of an IR phase-modulated ellipsometer for the in situ characterization of surfaces and thin films (*E3*). Kobayashi et al. (*E4*) employed in situ reflection/absorption spectroscopy to investigate the surface reactions in selective chemical vapor deposition (CVD) of tungsten using tungsten hexafluoride and silane. Molecular interactions between organized, surface-confined monolayers and vapor-phase probe molecules were studied by in situ external reflectance spectroscopy and discussed by Xu and co-workers (*E5*). The microscopic interactions between an organic monolayer and contacting liquids were studied by IR/visible sum-frequency spectroscopy (SFS) by Ong and co-workers (*E6*). Honda and co-workers (*E7*) described the design and implementation of an apparatus for the in situ measurement of IR absorption and emission spectra of oxide films formed on chromium metal at temperatures up to 973 K. A new experimental setup was described by Persson and Leygraf for the quantitative measurement of atmospheric corrosion of metal surfaces (*E8*). Nishida and co-workers investigated the oxidation of silicon surfaces in CVD studies using sensitive in situ IR measurements (*E9*). Several IR approaches were evaluated by Armstrong et al. (*E10*) for the study of arsine decomposition in order to investigate gallium arsenide film production. Kellar and co-workers reported the in situ IR study of double-bond reactions of adsorbed oleate at a fluorite surface (*E11*). The reaction process of tungsten hexafluoride with hydrogenated amorphous silicon was studied by polarization modulation IR spectroscopy and quadrupole mass spectrometry (*E12*). Morrison and Haigis conducted in situ IR studies during the plasma deposition of amorphous silicon (*E13*). The in situ measurement of CVD titanium oxide films was performed by Wadayama and co-workers using double-modulation IR spectroscopy (*E14*). Blayo and Drevillon studied the early stage of the growth of plasma-deposited amorphous silicon on glass substrates by in situ IR phase-modulated ellipsometry (*E15*). Pigeat and co-workers reported the use of IR emission spectroscopy for the investigation of the porosity of thin oxide films (*E16*). The role of hydrogen in surface reactions of amorphous silicon was investigated by Kawamura and co-workers using ATR spectroscopy (*E17*).

Chemicals and Polymer Studies. Marand and co-workers conducted in situ IR experiments and measured microwave dielectric properties to compare the reaction mechanisms of epoxy resins undergoing thermal and microwave curing (*E18*). The kinetics of CN bond transformation into a conjugated CN bond in acrylonitrile copolymer was studied by Zhao and co-workers using in situ IR measurements (*E19*). The processes of sorption/desorption of 1,2-dibromoethane on clay mineral surfaces was investigated by Aochi and co-workers using in situ IR spectroscopy (*E20*). Mu and Malhotra explored a new approach in the investigation of coal/water interactions by studying the O–H stretching region using in situ IR spectroscopy (*E21*). Farquharson and Chauvel discussed their kinetic studies of phosgene reduction performed by in situ IR measurements (*E22*). The cure cycle of PMR-15 was studied by Parker and co-workers (*E23*) using in situ IR measurements. Green and co-workers evaluated the use of long-path IR measurements for the determination of methyl bromide in indoor air (*E24*). A deep-immersion probe, designed for mid-IR measurements, was applied to the study of a polymerization reaction and reported by Doyle (*E25*). Tan and Arnold reported the synthesis and characterization of poly(amic dialkylamides) impregnated with thermosettable secondary amines, studied by in situ IR spectroscopy (*E26*). The in situ monitoring of extraction and separation behavior of lipids with supercritical carbon dioxide using IR spectroscopy was discussed by Ikushima et al. (*E27*). Martin and co-workers performed in situ IR studies of rubidium- and potassium-doped fullerenes in order to investigate and assign the vibrational modes (*E28*). The change of conformational disorder in membrane lipids in the pulmonary artery of monocrotaline-injected rats was detected by in situ IR spectroscopy and reported by Yoshida and co-workers (*E29*). Kandori and Ishikawa investigated the effects of water on dispersion stability of colloidal particles in nonpolar organic media by IR spectroscopy (*E30*). Patel and co-workers used IR spectroscopy to sudy the kinetics of the thermal conversion of poly(phenylene-1,2-dibromoethylene) to poly(phenylene-acetylene) (*E31*).

Electrochemical Studies. The in situ spectroscopy of polyaniline in aqueous solutions was investigated by Moser et al. using IR/ATR spectroscopy with the metal grid evaporated directly onto the surface of the zinc selenide ATR crystal (*E32*). Ionic adsorption at the solid/solution interphase was investigated using three in situ methods, including IR spectroscopy (*E33*). IR spectroscopy was used by Parry et al. to study the spectroelectrochemistry of bisulfate and sulfate adsorption on gold with and without the underpotential deposition of copper (*E34*). In situ IR was used by Widder and co-workers (*E35*) to study the lithium electrode surface in a model lithium battery by internal reflection spectroscopy as well as by photothermal beam deflection. Trettenhahn and co-workers performed an in situ investigation of lead electrodes in 5 M sulfuric acid in order to study the formation and consumption of lead sulfate (*E36*). Electrochemical processes at the silicon/acetonitrile interface were investigated by Boonekamp et al. using IR spectroscopy (*E37*). Russell and co-workers investigated the potential dependence of silver/water interactions using in situ IR spectroscopy (*E38*). Lin and Li described the construction and utilization of a cell designed for analysis by spectroelectrochemistry in the IR at micro- and ultramicroelectrode surfaces (*E39*). Kvarnstrom and Ivaska described the in situ external reflection IR studies on electrochemically polymerized polyphenylene (*E40*). Lin and co-workers reported on the quantum chemistry and IR studies of carbon monoxide on platinum electrodes (*E41*). Bockris and Jeng investigated the adsorption of organic compounds on platinum electrodes by IR spectroscopy (*E42*). IR studies of the electrochemical oxidation of adsorbed carbon monoxide on nickel in alkaline solutions were reported by Zhao and co-workers (*E43*). Christensen et al. reported the in situ IR study of the electroreduction of polybithiophene (*E44*). Scherson discussed the IR studies of transition metal macrocycles as catalysts for the electrochemical reduction of oxygen (*E45*). Jones and Hinman reported the studies of tetraphenylporphyrin complexes containing manganese, iron, and cobalt using in situ IR spectroscopy (*E46*). Chazalviel and co-workers investigated the audiofrequency modulation of the electrode potential together with IR spectrocopy for the study of electrochemical interfaces (*E47*). The study of the ruthenium electrode in acid and alkaline solutions was performed using in situ IR spectroscopy and reported by Bewick and co-workers (*E48*). The same authors also investigated the anodic oxide film on cobalt in alkaline solutions (*E49*). Faguy and co-workers studied the adsorption of acetonitrile on a gold electrode from aqueous solutions using IR spectroscopy (*E50*). The spectroscopic studies of redox-active self-assembled monolayers on gold electrode surfaces were reported by Bae and co-workers (*E51*). Iwasita and Nart identified the forms of methanol adsorbates on platinum using in situ IR spectroscopy (*E52*). The study of the adsorption geometry of bisulfate ions on a platinum electrode was reported by Samant and co-workers (*E53*). Christensen et al. investigated charge conduction in polybithiophene using in situ IR spectroscopy (*E54*). Spectroelectrochemical studies of cyanide adsorbed on platinum and palladium were reported by Ashley et al. (*E55*). Ichino and co-workers (*E56*) investigated the poly(ethylene oxide)/lithium perchlorate metallic lithium interface using in situ IR spectroscopy. Novak et al. reported the study of the overoxidation of polypyrrole in propylene carbonate by IR spectroscopy (*E57*). In situ IR and mass spectrometries were used by Cattaneo and co-workers (*E58*) to study the anodic stability of propylene carbonate electrolytes at potentials above 4 V against lithium. Pham et al. (*E59*) described the IR studies of the electrochemical immobilization of heteropolyanions in poly(1-naphthol)-coated electrodes. A novel optically transparent thin-layer electrode for in situ spectroelectrochemical studies was described by Xiao and co-workers (*E60*). Stole reported the characterization of mono- and multimolecular assemblies at glassy carbon and noble metals by ex situ and in situ IR external reflection spectroscopy (*E61*). Talonen and co-workers studied the adsorption of ethyl xanthate on gold, silver, and copper electrodes under contolled potential (*E62*). Mechanistic studies of the electrochemical doping/undoping process of poly(1-naphthol) film using IR spectroscopy were reported by Pham and Moslih (*E63*). The influence of water on the oxidation of propylene carbonate on platinum was studied by Rasch and co-workers (*E64*). Sariciftci and co-workers investigated the structural mechanism of a zwitter-viologen system during electrochemical charge-transfer reactions (*E65*). Bae et al. (*E66*) studied the oxidation of glucose on platinum in alkaline media. Srinivas and co-workers performed an in situ IR study coupled with transient analysis of hydroformlyation (*E67*). Carbon monoxide adsorbed at iridium(111)/aqueous interfaces was studied with respect to the double-layer effect on the adlayer structure by Jiang and co-workers (*E68*).

Catalyst Studies. Carbon monoxide oxidation over palladium and copper catalysts was investigated with respect to alumina-supported bimetallic palladium–copper particles using in situ IR spectroscopy by Choi and Vannice (*E69*). Peden and Hoffman (*E70*) studied the 2140-cm^{-1} carbon–oxygen stretching vibration in order to investigate the carbon monoxide oxidation reaction over ruthenium(001). The IR study of carbon monoxide/hydrogen reactions over ruthenium/silica at high pressures and temperatures was reported by McQuire and Rochester (*E71*). The investigation of intrazeolite carbonyl metal chemistry by in situ diffuse reflectance IR measurements was described by Boulet-Desgrousilliers and co-workers (*E72*). Dehydration, deammoniation, and reammoniation of ion-exchanged zeolites were studied using IR spectroscopy from 40 to 4000 cm^{-1} by Jacobs and co-workers (*E73*). McQuire et al. reported the investigation of carbon monoxide/hydrogen reactions over rhodium/alumina catalysts at high pressure and temperature (*E74*). Similar research over ruthenium–rhodium/silica catalysts was performed by McQuire and Rochester (*E75*). Bache and Ystenes developed a cell for the study of different reactions in Ziegler-Natta catalyst systems (*E76*). IR and flow reactor studies on heterogeneously catalyzed gas-phase ammonimation of cyclohexanone were conducted by Dreoni and co-workers (*E77*). The influence of vanadium(V) on the thermal stability of 12-metallophosphoric acids was studied using in situ IR spectroscopy in the investigation of catalysis by polyoxometalates by Rocchiccioli-Deltcheff and Fournier (*E78*). Gargulak and co-workers investigated the homogeneous catalytic carbonylation of nitroaromatics using in situ high-pressure IR studies (*E79*). The formation of benzene-adsorbed species from acetylene over a titanium dioxide catalyst was observed in in situ IR studies by Sakata et al. (*E80*). Pien and Chuang performed in situ IR studies of ethylene hydroformylation and carbon monoxide hydrogenation on ruthenium/silica and sulfided Ru/SiO$_2$ (*E81*). Analysis of the surface species formed during the methylation of toluene over HZSM-5 zeolite was performed by Mirth and Lercher (*E82*) using in situ IR spectroscopy. In situ IR was used to study the diffusion of benzene and ethylbenzene in ZSM-5-type zeolites by Niessen and Karge (*E83*).

(F) ON-LINE

In-line analysis of polymer composition in polymer blends and yellowness index of polymers were discussed as measurements which can be performed by fiber-optic near-IR spectroscopy by Mc Peters and Williams (*F1*). The feasibility of on-line monitoring of the Btu content of natural gas with a near-IR fiber-optic analyzer was investigated by Brown and Lo (*F2*). The application of near-IR reflectance spectroscopy to the on-line analysis of cellulose esters was demonstrated by De Wit (*F3*) using a multiple filter-based instrument. Lange and co-workers were awarded a U.S. patent for the process control method of on-line vibrational spectroscopy for the production of polyolefins (*F4*). Zetter and Politzer described the application of near-IR spectroscopy on-line for the measurement of octane number in gasoline blending (*F5*). Coates and Reber described the development and implementation of a near-IR analyzer for on-line monitoring of liquid streams such as in a chemical processing environment or oil refining application (*F6*). Nivens and co-workers (*F7*) demonstrated the utility of a multichannel ATR/FT-IR spectrometer for the on-line examination of microbial biofilms. The quantitative monitoring of 6-aminopenicillanic acid production by on-line FT-IR spectroscopy was demonstrated by Guzman and co-workers (*F8*). Ghosh described an on-line near-IR technique for controlling the application of poly(vinyl alcohol) sizing to cotton warp (*F9*). Fidler reported the use of on-line FT-IR measurements for the analysis of additive levels in polymer melts (*F10*). Jones and co-workers described the use of transient IR emission spectroscopy in order to obtain useful spectra from opaque moving solids, such as the cure of an acrylic coating on a polycarbonate substrate (*F11*). The use of an FT-near-IR spectrometer and optical fibers was applied to the on-line analysis of a set of aliphatic alcohol mixtures by Mackison and co-workers (*F12*). Grob et al. (*F13*) demonstrated the utility of near-IR fiber-optic transmission spectroscopy for the on-line monitoring of polyol production. The development and use of an on-line IR analyzer system to monitor cephamycin C loading on an ion exchange resin was reported by Shank and co-workers (*F14*). The use of near-IR spectroscopy in the range 700–1300 nm was described by Chen and Feng for the determination of gasoline distribution characteristics (*F15*). Christy and co-workers described the use of diffuse reflectance IR spectroscopy for the on-line monitoring of the pyrolysis of geochemical samples (*F16*). A chemometric methodology for fixed-filter IR analyzer specification was developed and reported by Moessner and Russell (*F17*). The utility of near-IR analyzers for real-time process control was also discussed by Moessner (*F18*). The design and utility of a high-temperature, high-pressure flow cell for the on-line IR analysis of molten polymers was reported by Fidler and co-workers (*F19*). Stojkovic et al. (F20) demonstrated the utility of IR spectroscopy for monitoring the process of crystallization and the determination of the degree of crystallinity of NaA zeolites. Fiber-optic evanescent wave spectroscopy using IR-transparent optical fibers and tunable lead salt lasers was described by Bunimovich (*F21*). Mc Dermott (*F22*) described the use of near-IR spectroscopy for on-line blending control for beer production. Cermelli and co-workers (*F23*) reported the use of on-line near-IR spectroscopy for various applications in the petrochemical industry such as steam cracking and manufacture of polyethylene and polyisobutene. The design and use of a multipass cell coupled to a combustion flow reactor was reported by Koshland and co-workers for the measurement of the efficacy of thermal destruction of chlorinated hydrocarbons (*F24*). Hammond and Brookes discussed the use of near-IR spectroscopy for the analysis of fermentations (*F25*). Driver described some environmental considerations for the use of fiber-optic remote sensing systems for on-line process monitoring (*F26*). An on-line wastewater analyzer for monitoring organic pollutants in hazardous waste streams using FT-IR spectroscopy with a sampling and sparging system was discussed by Mc Intosh and co-workers (*F27*). The determination of on-stream toxic air emissions using FT-IR spectroscopy was reported by Wentz et al. (*F28*). An on-line analyzer system using a process gas chromatograph and an IR analyzer was used by Stephanos and Lamb (*F29*) to monitor carcinogenic and toxic components in ambient air. Wuelbern investigated the use of an FT-IR spectrometer for on-line monitoring of flue gases of a modern bituminous coal-fired heating power plant (*F30*). Green and co-workers reported on the investigation of long-path IR measurements for the determination of volatile toxic organics in indoor air (*F31*). Stuart discussed the use of IR gas filter correlation detection for the quantitative detection of a number of common industrial stack gases (*F32*). The design and use of a piezo-enhanced multireflection cell for trace measurements of gases was reported by Kronfeldt and Berger (*F33*). The development and use of an in situ, dispersive interferometer for the measurement of thermal efficiency and the effluent flow rates of fossil-fired boilers was reported by Shlifshteyn and Lang (*F34*). A portable long open path FT-IR system for the measurement of trace gases in ambient air pollution was discussed by Tso et al. (*F35*). Fleming and co-workers described the monitoring system for the measurement of carbon tetrachloride and chloroform in wastewater based on continuous closed loop sparging and FT-IR vapor measurement (*F36*).

(G) ENVIRONMENTAL ANALYSIS

Kricks et al. (*G1*) demonstrated the field capability of using open-path FT-IR spectroscopy to monitor the air surrounding a Superfund Landfill site. Russwurm et al. (*G2*) used a Fourier transform infrared spectrophotometer to measure chemical emissions at the Shaver's Farm Superfund site in northwestern Georgia. Average concentrations of target gases were inferred by matching measured spectra with reference spectra of precisely measured quantities of the target gases. Kasper et al. (*G3*) used portable IR spectroscopy to measure the total petroleum hydrocarbons in soils. More than 600 surface and subsurface solid samples from several sites were analyzed for fuel-related hydrocarbons. Litzenberg et al. (*G4*) determined total hydrocarbon content of contaminated soils using a portable infrared analyzer and found results to be within 1 order of magnitude with certified laboratory results. Fernandez (*G5*) used aerial IR survey techniques to survey hydrocarbon contamination of soils. The survey results confirmed the placement of a test well, along with several areas of heavy concentration of organic material, and movement from the product saturated areas to the recovery system.

Andrew (*G6*) determined nonionic surfactants in wastewater at the 0.1–50 mg/L level using direct extraction and monitoring the 1110-cm^{-1} absorbance band by FT-IR. Thurston et al. (*G7*) identified alkyl and chloroalkyl phosphates in water effluent from a plant manufacturing fire-retardant chemicals using low- and high-resolution mass spectrometry and FT-IR. Whitcraft and Wood (*G8*) monitored CH_2Cl_2, MeOH, NH_3, and SF_6 in plume emissions from a surface-aerated equalization basin of a wastewater treatment plant. Kagann and Simpson (*G9*) used a remote FT-IR sensor to measure air pollution from a chemical wastewater treatment plant. Capri et al. (*G10*) determined isocyanates in water samples by IR spectrophotometry. Arryanto and Bark (*G11*) precipitated trace quantities of poly(acrylamide) in potable water with reineckate ions. The poly(acrylamide) concentrations were obtained by determining the Cr content of the precipitate by atomic absorption spectrometry or by using IR reflectance spectrometry. Krskaa et al. (*G12*) used fiber-optic evanescent wave spectroscopy based on AgClBr fibers and FT-IR to determine chlorinated hydrocarbons in water. A minimum detection limit lower than 10 mg/L was achieved by coating the fiber with low-density polyethylene, which showed reversible enrichment of the chlorinated hydrocarbon. Richardson et al. (*G13*) used gas chromatography coupled with low- and high-resolution electron-impact mass spectrometry, low and high chemical ionization mass spectrometry, and FT-IR to identify eight straight-chain aldehydes in a water sample taken from the overflow pipe at a municipal sewer line containing both industrial and domestic sewage. Doyle (*G14*) used sparging FT-IR to monitor trace concentrations of contaminants in water down to the low ppb range for many aromatics and chlorinated hydrocarbons.

Wang et al. (*G15*) used a remote FT-IR spectrometer to measure MeOH, CO, and CO_2 in motor boat exhaust. Herget and Lowry (*G16*) monitored either diluted or raw exhaust gas using a 20-m multiple-pass cell and FT-IR spectroscopy. Measurements were taken at ambient temperature or at 100 °C. Classical least-squares fitting was used in the analyses. Minimum detectable concentrations are dependent on cell volume, gas flow rate, measurement time resolution, and spectral resolution. Axelsson et al. (*G17*) described the construction of a long-path FT-IR instrument that can be used to measure volatile organic compounds emitted from an automobile painting facility. Microgram levels of aromatic hydrocarbons were detected along a 350-m folded measurement path using 180 scans at 1 cm^{-1} resolution. Arai (*G18*) used FT-IR to continuously measure unregulated emission from diesel engines. Improvements in measuring the exhaust were made by heating the gas sampling line, filtering particulate material, and modifying the window material of the sampling cell. HCHO and NH_3 as well as regulated pollutants could be monitored.

Demirgian et al. (*G19*) discussed applications of using conventional FT-IR for continuous monitoring of incinerator emissions and determining indoor air quality. Applications of conventional and passive-remote FT-IR are discussed.

Passive-remote FT-IR can be used to identify and track chemical plumes along with fugitive emissions. Kasai et al. (*G20*) used FT-IR along with a moisture exchanger made of a molecular exchange membrane to determine concentrations of NO_x every 3 s in combustion gases of fossil fuels without interference from water vapor and other vapors. Lord and Brown (*G21*) used an open-path monitor, based upon a nondispersive IR analyzer with a solid-state detector, to monitor toxic combustible or hazardous vapors. This allowed ppb real-time sensitivities to be obtained when an extended path length was used. Russwurm et al. (*G22*) used long-path FT-IR to determine volatile organic compounds in New Castle, DE, as part of the Superfund Innovative Technology Evaluation program. The data were compared to data taken with a whole-air canister technique. Brandon and Trautwein (*G23*) monitored the quality of ambient air in workplaces using remote sensing FT-IR. MeOH and Freon-12 were determined in an electronics assembly area, while HF was monitored in an aluminum smelter workplace. Foosnaes et al. (*G24*) describe a dedicated FT-IR combined with a PC or PL interface to continuously monitor gaseous fluoride emission from aluminum reduction cells. Franzblau et al. (*G25*) reported using a transportable FT-IR spectrometer for determining MeOH vapor in alveolar and ambient air. The data suggest that FT-IR spectroscopy is a practical and efficient approach for simultaneous biological and area monitoring of human exposure to organic solvents. Johansson and Brunstroem (*G26*) evaluated the performance of a multicomponent IR spectrometer designed to measure NO, NO_2, CO, CO_2, N_2O, NH_3, and H_2O, with regard to possible interferences from compounds present in flue gas. Phillips et al. (*G27*) describe a high-speed multizone IR absorption spectrometer for tracer gas ventilation experiments. The approach used IR absorption to measure concentrations of SF_6, which was seeded into the enclosure. The apparatus took simultaneous, continuous concentration measurements. Accuracy of a relative concentration measurement was better than 1.0%, while absorbance measurements were accurate to 5.0%. Ter Kuile et al. (*G28*) discussed using a IR gas cloud scanner developed for identification of hazardous gases in the work environment. The scanner provided fast, remote, and quantitative identification of gases by imaging gas dispersion in the work environment. This information can be effectively used in helping to improve noncompliance in work situations by providing information on causes and gas dispersion patterns. Hudson et al. (*G29*) determined that a field mobile open-path FT-IR remote sensing instrument was effective in monitoring targeted toxic air pollutants at a chemical facility. CH_2Cl_2 concentrations were found to be 5.6–31.6 ppb in a 51-min period that coincided with a uncontrolled release of chloromethanes. Xiao et al. (*G30*) describe a transportable remote sensing instrument that is capable of performing real-time quantitative analysis of gas and vapor contaminants in air in the workplace. A method was also developed to overcome the effect of nonanalyte compounds present in the background spectrum on the quantitation of targeted analytes in the sample spectrum. Kirchgessner et al. (*G31*) developed a procedure to determine methane emissions from surface coal mines using open-path FT-IR spectroscopy and modeling techniques. Results revealed that emissions from one surface coal mine in the United States were 1 735 000 m^3/yr. The results, however, provide some evidence that methane concentrations determined by IR may be low by 20–75%. The trial did demonstrate that the method is applicable and feasible for measuring such emissions from very large surface coal mines.

Fateley et al. (*G32*) used long-path FT-IR to detect emission of volatile organic compounds over wastewater treatment facilities. Green et al. (*G33*) monitored postfumigation indoor air for MeBr and chloropicrin down to the ppb level (laboratory test data) using an FT-IR spectrophotometer interfaced to a 2.5-m base path, open multiple reflection cell. Palen et al. (*G34*) determined the chemical composition of smog chamber aerosol generated during the photooxidation of isoprene and (−)-pinene using FT-IR microspectroscopy. Lee and Van Ewyk (*G35*) used an IR detector having a first source of light at the focal point of a first off-axis reflector, a second source of light at the focal point of a second off-axis parabolic reflector, and a beam-combining filter to combine the collimated beams from the two sources to detect toxic or combustible gases in air of coal mines, oil platforms, or chemical plants. Simpson (*G36*) described a method for detecting the presence of a combustible IR absorbing gas in an air atmosphere located in hazardous locations such as oil and gas rigs. Plummer (*G37*) used FT-IR to determine eight toxic compounds taken from the inlet and outlet of a gasoline vapor control device at a bulk-loading gasoline facility. The compounds were determined in charcoal trap samples and compared to gas chromatography flame ionization detector results.

Taylor (*G38*) determined the spectral response in the visible and near-IR of different amounts of crude oil on water. Spectral reflectance measurements were made of selected beaches and beach materials to provide ground truth data for the MEIS II imagery collected during the *Exxon Valdez* spill. Lin and Brown (*G39*) used near-IR spectroscopy based on perturbations of water bands by sea salts. Models expressing salinity were developed by linear or mutilinear regression of absorbancies at selected wavelengths and by principal component regression using the entire spectrum. The technique can potentially be used in remote sensing of seawater salinity.

Massie et al. (*G40*) determined the absorption cross sections at 93, 273, 253, 233, 213, and 203 K for various chlorofluorocarbons. The cross sections were displayed in graphical form to facilitate quick identification and quantification of CFC gases in high- and medium-resolution sensing studies.

Tuchman (*G41*) used FT-IR to determine quartz in respitable dust trapped on filters. Bell et al. (*G42*) found that diethyl ether interferes with infrared breath analysis for ethanol. Shreve et al. (*G43*) used near-IR as an alternative technique which provides an estimate for the determination of nicotine in cigarette smoke presented to animals in inhalation studies.

Stor-Pellinen et al. (*G44*) reported preliminary results of a study to detect impurities in recycled paper, glass, and chipped wood used for pulp production by monitoring IR emissions, and the possibility of categorizing collected plastics by IR imaging.

(H) FOOD ANALYSIS

Infrared spectroscopy, especially in the near-infrared, continued to be useful in the analysis of foods. Ismail et al.

(*H1*) used IR both in transmission and ATR to quantitatively determine free fatty acids in fats and oils. The degree of unsaturation in edible oils was measured by Ulberth and Halder (*H2*) and by Afran and Newbery (*H3*) using ATR spectroscopy. The nondestructive determination of oil content in single kernels of maize was performed by Orman and Schumann (*H4*).

Van de Voort et al. (*H5*) developed a quality control method for fat and moisture determination in butter. Ozanich et al. (*H6*) performed a noninvasive determination of the moisture and oil content of wheat flour cookies. The crude lipid content in the muscle of intact rainbow trout was measured by Lee et al. (*H7*) and in salmon by Sollid and Solberg (*H8*). The protein content in oil/water emulsions was studied by Kamishikiryo et al. (*H9*). Protein, fat and water in plastic wrapped, homogenized meat were measured by Isaksson et al. (*H10*). Physical and chemical characteristics of beef cuts were determined by Mitsumoto et al. (*H11*).

Near-IR analysis of sugars in sugar cane juice, raw sugar, and bagasse was performed by Clarke et al. (*H12*). Mixtures of sugars in solution were quantitatively determined by Kemsley et al. (*H13*). Dupuy et al. (*H14*) determined sugars and organic acids in dry extracts of fruit juices. A correction for the interference of limonene in the determination of citral in lemon and orange essential oils was reported by Lopez Mahia et al. (*H15*).

Van de Voort et al. (*H16*) assessed the use of IR spectroscopy for the analysis of milk. The influence of highly unsaturated milk fat on the accuracy of milk fat analyses was discussed by Stegeman et al. (*H17*). Downey et al. (*H18*) used near-IR to classify skim milk powders. Alternative calibration procedures for IR milk analyzers were investigated by Hill et al. (*H19*). A procedure for the quantitative determination of fat, protein, lactose, and water in cow milk was described by Luinge et al. (*H20*). The influence of homogenization efficiency on milk fat content determination was studied by Remillard et al. (*H21*).

Williams et al. (*H22*) analyzed oat bran products using near-IR reflectance spectroscopy. Estimation of polysaccharides in barley was made by Szczodrak et al. (*H23, H24*). Delwiche et al. (*H25, H26*) correlated moisture content and temperature with water activity in starch and cellulose. A procedure for the assay of *S*-methylmethionine in malt was reported by Yan et al. (*H27*). Biochemical changes in malting were monitored by Allison and Maule (*H28*). Lebouille and Drost (*H29*) used partial least squares to analyze barley and malt parameters. Alcoholic fermentation in wines was followed by Davenal et al. (*H30*). Ethanol was determined in alcoholic beverages by Lopez et al. (*H31*).

(I) BIOCHEMICAL APPLICATIONS

Infrared spectroscopy continued to be utilized extensively in the analysis of biological molecules. A review article detailing advances in instrumentation and data processing for Fourier transform infrared spectroscopy and its application to complex biological systems was presented by Jackson and Mantsch (*I1*).

Near-IR spectroscopy was used to determine pH, glucose, and serum albumin concentrations in a simulated biological matrix in experiments by Drennen et al. (*I2*). The amide I bands of α-lactalbumin were examined by Prestrelski et al. (*I3*). McFarlane et al. (*I4*) studied the components of the secondary structure of the human parathyroid hormone-related protein by infrared spectroscopy. Human ceruloplasmin was characterized by Haris et al. (*I5*). White et al. (*I6*) measured the contribution of protein perturbation in chymotrypsin ester carbonyl groups. The incorporation of ion channel-forming peptides into Langmuir–Blodgett films of phosphatidic acid was studied by Lukes et al. (*I7*). Three-dimensional doorway-state theory was applied to analyze the structure and spectrum of the amide-I bands of globular proteins by Torii and Tasumi (*I8*). The amide I/amide II ratio in proteins in solution and in the solid state was compared using transmission, attenuated total reflectance (ATR) and diffuse reflectance spectrometries in a study by Ishida and Griffiths (*I9*).

The conformations of enzymes in the lyophilized and aqueous states was examined by Prestrelski et al. (*I10*). Kugo et al. (*I11*) used ATR to study the secondary structure of poly(γ-methyl L-glutamate) surfaces treated with formic acid. Ultrafast studies of protein dynamics were performed by Hochstrasser (*I12*). A general discussion of the use of FT-IR to study the secondary structure of proteins was prepared by Mantsch and Surewicz (*I13*). The secondary structure of proteins and polypeptides in halogenated alcohols was examined by Jackson and Mantsch (*I14*). Self-association of uracils in argon matrices was studied by Maes et al. (*I15*). Infrared studies of the β-sheet structures in connectin fibers were pursued by Uchida et al. (*I16*). Carmona et al. (*I17*) observed interactions between poly(L-asparagine) and L-aspartic acid with polynucleotides. Torii and Tasume (*I18*) performed model calculations on amide-I bands of globular proteins, comparing the results to experimental observations.

Infrared spectra of secondary structures of two recombinant murine proteins were compared to results obtained by other analytical techniques, emphasizing the need for a variety of characterization procedures in a study by Wilder et al. (*I19*). Abbott et al. (*I20*) analyzed jojoba protein conformations in water. Goormaghtigh et al. (*I21*) used ATR to study the secondary structure of the membrane-bound form of the pore-forming domain of colicin A. Jackson and Mantsch (*I22*) correlated IR spectral information about protein secondary structures with three-dimensional Ramachandran plots. Quantitative information about the secondary structure of soluble and membrane proteins was obtained by Lee et al. (*I23*) using factor analysis techniques. Changes in proteins and polysaccharides in a microbial biofilm were monitored with ATR by Bremer and Geesey (*I24*). Artifacts in proteins in solution were presented by Jackson et al. (*I25*). Elucidation of the structures of calcium-binddioxygenase was investigated by Uchida et al. (*I26*). Haris et al. (*I27*) employed carbon-13 and nitrogen-13 labeling to study protein/protein interactions in two bacterial proteins.

The relationship between the structure and binding characteristics of rat liver L-tryptophan associated with the determination of protein secondary structure using ATR spectroscopy were discussed by Jackson and Mantsch (*I28*). Structural differences between type I and type IV collagen in biological tissues measured in vivo using ATR spectroscopy were evaluated by Osaki et al. (*I29*). Fabian et al. (*I30*) examined the secondary structure of streptokinase in aqueous solution. Yang (*131*) discussed the development of a method for estimating the secondary structure of proteins from IR spectra obtained in aqueous solution. The conformation of magainin-2 in aqueous solution was investigated by Jackson et al. (*132*). Singh et al. (*133*) tested the sensitivity limits for the measurement of protein secondary structure by ATR spectroscopy.

Mantsch et al. (*134*) characterized β-turns in cyclic hexapeptides in solution. Folding in protamine was investigated by Pesek and Shabary (*135*). Infrared and NMR spectroscopies were used by Gerothanassis et al. (*136*) to study hydration of the Gly2 and Gly3 peptide oxygens in [Leu5]-enkephalin in aqueous solution. Pressure-induced secondary structures of five globular proteins were studied by Taniguchi and Takeda (*137*). Prestrelski et al. (*138*) generated a substructure library for the description and classification of protein secondary structures. Cho et al. (*139*) applied near-IR spectroscopy to the on-site determination of impairment of amino acid residues caused by reaction with carbonyl compounds. Zuber et al. (*140*) commented on the application of infrared spectroscopy to studies of aqueous protein structures.

The role of hydrogen bonding in the catalysis of deacylation in acylchymotrypsin was investigated by White et al. (*141*). Tonge and Carey (*142*) developed a quantitative measure for the degree of ground-state destabilization in the reaction of serine proteases. Wong (*143*) studied H/D exchange kinetics in chymotrypsin at various pressures. Ismail et al. (*144*) investigated changes in the secondary structure and aggregation of chymotrypsinogen.

Ultrafast IR studies of protein dynamics were performed by Deller et al. (*145*). The secondary structure and conformation of bovine lens proteins was examined by Lamba et al. (*146*). IR spectra of bovine lens crystallins in the solid state were recorded by Rozyczka and Gutsze (*147*). Resonance Raman and IR spectra of retinal proteins were described by Siebert (*148*). The secondary structure of bovine P2 protein in deuterium oxide was investigated by Stuart and McFarlane (*149*). Secondary structures of bovine insulin and its derivatives were discussed by Wei et al. (*150*).

Infrared spectra of glucose complexes with alkaline earth metals were studied by Sharareh and Wilkins (*151*). Nonlamellar structures of lipopolysaccharides were investigated by Brandenburg and Seydel (*152*).

Vibrational spectroscopy and circular dichroism (CD) spectroscopy were used by Carmona et al. (*153*) to determine the conformational structure of an L-asparagine/L-aspartate random copolypeptide. Pancoska et al. (*154*) examined the vibrational CD (VCD) spectra of globular proteins in deuterium oxide solution. FT-IR and CD spectra were used by Perczel et al. (*155*) to characterize peptide conformations. Experimental and computational peptide conformations were compared in studies by Birke et al. (*156*). Wyssbrod and Diem (*157*) obtained vibrational CD spectra of peptide β-turns in a cyclic pentapeptide. Pietrzynski et al. (*158*) investigated conformations of model peptides in solution. The amide III VCD signals of peptides were interpreted by Birke et al. (*159*). Gulotta et al. (*160*) observed conformational changes in DNA molecules with VCD. The temperature dependence of the secondary structure of troponic C from bullfrog skeletal muscle was measured by Yamamoto et al. (*161*). Paterlini et al. (*162*) performed VCD studies of interchain hydrogen-bonded tripodal peptides.

A thesis by Markovich (*163*) featured the use of FT-IR to study immobilized and mobile artificial membranes. Haris and Chapman (*164*) discussed the use of infrared spectroscopy as a probe of the study of secondary structures of membrane proteins. Coherent nuclear motion in a membrane protein using femtosecond near-IR spectroscopy was monitored by Vos et al. (*165*). Auger (*166*) published a thesis studying anesthetic/lipid interactions in lipid bilayers. The interaction of toxic metal ions (Cd, Hg, Pb) with chloroplast thylakoid membranes was studied by Ahmed and Tajmir-Riahi (*167*). Site-directed mutagenesis was used to assign infrared bands to amino acid residues in studies by He et al. (*168*). Zhang et al. (*169*) studied conformation and amide hydrogen exchange of membrane proteins. Quantitative determination of conformational disorder in membranes was studied by Mendelsohn (*170*). Baenziger et al. (*171*) probed conformational changes in membranes of nicotinic acetylcholine receptor. Fernandez-Ballester et al. (*172*) studied protein stability and interaction of membranes of the nicotinic acetylcholine receptor with cholinergic ligands.

A general survey of infrared spectroscopic characterization of DNA was written by Taillandier and Liquier (*173*). Hydration-driven conformational changes of DNA were monitored in studies by Pohle (*174*). Maleev et al. (*175*) studied the formation of the DNA helix structure and the role of its hydration environment on double-helix stabilization energy. Taillandier (*176*) studied triple-helical DNA structures. Saxena and Van Zandt (*177*) investigated the effect of counterions on the spectrum of dissolved DNA polymers. Interactions of nucleic acids and halo compounds were studied by Bottura et al. (*178*). Adnet et al. (*179*) studied the interactions between polynucleotides and minor groove binding compounds. The influence of protein/nucleic acid ratio on the vibrational spectra of nucleoproteins was investigated by Litvinov et al. (*180*). Experimental spectra of DNA and RNA viruses were compared with computer models by Belokour et al. (*181*). The secondary structure of a *Bacillus licheniformis* endo-1,3–1,4-glucanase was measured by IR spectroscopy and compared to theoretical predictions by Querol et al. (*182*).

Molecular changes after oxidoreduction of cytochrome were characterized by Berthomieu et al. (*183*). Cyanide binding to bovine heart cytochrome *c* oxidase was examined by Tsubaki and Yoshikawa (*184*). Investigations into the structure of cytochrome *b*-559 were conducted by Berthomieu et al. (*185*). Redox-dependent changes in the secondary structure of cytochrome *c* in various animals were examined by Dong et al. (*186*). Yoshikawa and Caughey (*187*) exhibited infrared evidence for azide binding to metal and nonmetal sites in heart cytochrome *c* oxidase. Hill et al. (*188*) demonstrated the structure of b_o-type ubiquinol oxidase of *Escherichia coli* and the assembly mechanism of its binuclear center. The effect of cell differentiation in human leukemic cells and saphenous vein internal wall as induced by TPA enzyme was studied by Romano et al. (*189*) using ATR and microspectroscopies. The effect of surfactants on the structure of cytochrome *c* oxidase was studied by Castresana et al. (*190*). Cyanide binding to metal sites of bovine heart cytochrome *c* oxidase was investigated by Tsubaki and Yoshikawa (*I91*). Near-IR spectroscopy was used to determine cat brain cytochrome *c* oxidase after blood/perfluorocarbon exchange in experiments by Ferrari et al. (*I92*). The effect of crystallization on the heme–carbon monoxide moiety of bovine heart cytochrome *c* oxidase was studied by Tsubaki et al. (*I93*). The influence of temperature and pH on electrochemically induced conformational changes in cytochrome *c* was monitored by Schlereth and Maentele (*I94*). The dynamics of ligand binding to cytochrome *c* oxidase was investigated by Dyer et al. (*I95*).

The secondary structure of 5′-nucleotidase from bull seminal plasma was investigated by Fini et al. (*I96*). A kinetic analysis of alkaline phosphatase under hyperbaric manipulation was described by Wong and Armstrong (*I97*). Photodissociation of matrix-isolated respiratory enzymes from *E. coli* was studied by Hill et al. (*I98*).

Buchet et al. (*I99*) observed the effect of caged calcium release on the IR spectrum of sarcoplasmic reticulum vesicles. Barth et al. (*I100*) reported on the detection of small conformational changes in the catalytic cycle of sarcoplasmic reticulum ATPase. The thermotropic and conformational properties of a "hard-to-exchange" phospholipid population in the vicinity of calcium ATPase were investigated by Senak and Mendelsohn (*I101*).

Jayakumar et al. (*I102*) investigated the properties of monolayers of hydrophobic peptides in a lipid environment. The amide and acid bands of ganglioside GM1 were assigned by Mueller and Blume (*I103*). Deuterated segments of phospholipid molecules were used as structural probes in order to detect lipid phase transitions in specific membrane domains in a study by Torok et al. (*I104*). Diffuse reflectance FT-IR of derivitized lipid extract was used to distinguish pure cultures of archaebacteria from eubacteria by Hedrick et al. (*I105*). Pastrana et al. (*I106*) studied the secondary structure and orientation of a lung surfactant and its effect on the surface properties of phospholipids. The secondary structure of the surfactant protein SP-B in a lipid environment was investigated by Vandenbussche et al. (*I107*). Tanfani et al. (*I108*) examined the interaction of phospholipid bilayers with pig heart AMP deaminase. Jackson and Mantsch (*I109*) observed the interactions between valinomycin and ions in organic solvents, detergents, and lipid dispersions. Phase transitions in cuticular membranes, either as lipid extracts or lipids in situ, were studied by Gibbs and Crowe (*I110*). Cylindrical internal reflection was used by Iwaoka and Tabata (*I111*) to monitor lipid peroxidation in rat liver microsomes.

The structure and dynamics of melittin and analogs in lipid environments was studied by Weaver et al. (*I112*) using ATR spectroscopy. Frey and Tamm (*I113*) used polarized ATR to study the orientation of melittin in phospholipid bilayers. Ahmed et al. (*I114*) observed interactions of melittin with dimyristoylphosphatidylglycerol bilayers at various pressures. The effects of melittin and cholesterol on the molecular organization of phospholipid bilayers were investigated by Akyuz and Davies (*I115*). Membrane phase transitions in intact dry pollens with differential lipid compositions were investigated by Hoekstra et al. (*I116*). Infrared experiments used in the measurement of phase transition temperatures in membrane phospholipids were performed by Hoekstra et al. (*I117*). The effect of ganglioside GM1 on phospholipid membranes was studied by Mueller et al. (*I118*).

The structure and conformation of sphingomyelin was determined by Lamba et al. (*I119*) using IR and Raman spectroscopies. Evidence for alcohol binding and dehydration in phospholipid and ganglioside micelles was presented by Yurttas et al. (*I120*). Phase transitions in phosphatidylcholines from natural lipids were investigated by Morresi et al. (*I121*). The secondary structures of surfactant proteins in DPPC were investigated by Baatz et al. (*I122*). Surface composition at varying surface pressures in a model pulmonary surfactant was studied by Rana et al. (*I123*). The secondary structure of human low-density lipoprotein was discussed by Goormaghtigh et al. (*I124*). The conformational order in the acyl chains of living cell membranes of *Achoeplasma laidlawii B* was studied by Moore et al. (*I125*). Infrared spectra of cryoprotected lyophilized liposomes were discussed by Mobley and Schreier (*I126*). Castresana et al. (*I127*) studied the physical state of ubiquinone-10, in pure form and as incorporated into phospholipid bilayers. Conformational studies of high-density apolipoprotein discoidal complexes were performed by Yang et al. (*I128*).

An infrared kinetic study of the main phase transition of saturated phosphatidylcholine/water multilamellar systems was performed by Mellier et al. (*I129*). Naumann et al. (*I130*) studied phase transitions of single phosphatidylcholine bilayers on silica beads. The interaction of a synthetic model hydrophobic peptide with phosphatidylcholine bilayers was monitored by Zhang et al. (*I131*). Thermotropic phase transitions in egg yolk phosphatidylethanolamine were observed by Castresana et al. (*I132*). The effect of platelet-activating factor on dielaidoylphosphatidylethanolamine was studied by Salgado et al. (*I133*). The effect of cholesterol on the spectra of phosphatidylserines was investigated by Brumfeld et al. (*I134*). The effect of alcohols on hydrogen bonding of dipalmitoylphosphatidylcholine (DPPC) was studied by Chiou et al. (*I135*). Changes in physical properties with temperature in the liquid-ordered phase of mixtures of DPPC and cholesterol were monitored by Reinl et al. (*I136*). Studies of the interaction of deuterated DPPC with palmitic acid were performed by Villalain and Gomez-Fernandez (*I137, I138*). Structures of the subgel phases of *n*-saturated diacylphosphatidylcholine were examined by Lewis and McElhaney (*I139*). The effect of D-propranolol on dimyristoylphosphatidylcholine unilamellar vesicles was investigated by Cao et al. (*I140*).

Changes in the secondary structure of reconstituted nicotinic acetylcholine receptor were monitored by Gonzalez-Ros et al. (*I141*). Binding and interaction of carbamoylcholine with the nicotinic acetylcholine receptor were studied by Goerne-Tschelnokow et al. (*I142*). Spectroscopic evidence for protein structural changes upon desensitization of the nicotinic acetylcholine receptor was reported by Baenzinger et al. (*I143*). The secondary structure and thermal behavior of acetylcholine receptor membranes were investigated by Naumann et al. (*I144*).

Infrared spectroscopy was used to probe perturbations of the distal heme pocket in human myoglobin mutants in experiments by Balasubramanian et al. (*I145*). Deuterium bond variation in myoglobin was investigated by Le Tilly et al. (*I146*). The temperature dependence of the near-IR spectra of two heme proteins was studied by Leone et al. (*I147*). Models for heme d1 were analyzed by Mylrajan et al. (*I148*). The structure of the heme–copper binuclear center of the cytochrome b_0 complex of *E. coli* was studied by Tsubaki et al. (*I149*). Evidence for strong vibronic coupling in heme proteins was presented by Leone et al. (*I150*). Iron–carbon monoxide geometry in the subunits of carbonmonoxy hemoglobin M Boston was studied by Lian et al. (*I151*) using femtosecond IR spectroscopy. Near-IR was used to observe REM sleep-associated hemoglobin oxygenation in the monkey forebrain in a study by Onoe et al. (*I152*). A model for the major conformational substates in heme proteins was proposed by Oldfield et al. (*I153*). The interaction between heparin and poly(L-lysine) was examined by Grant et al. (*I154*). The immobilization process of heparin on polyethylene was monitored using ATR in experiments of Bertilsson et al. (*I155*) and Bertilsson and Liedberg (*I156*).

The structures of aspartic acid-96 in the L and N intermediates of bacteriorhodopsin were analyzed by Maeda et al. (*I157*). The mechanism of proton pumping of the M form of bacteriorhodopsin was investigated by Ormos et al. (*I158*). Kinetic studies of the L and M intermediates of bacteriorhodopsin were performed by Chen and Braiman (*I159*). Low-temperature studies of the L, M, and N photointermediates of bacteriorhodopsin were studied by Maeda et al. (*I160*). Transitions in the photocycle of bacteriorhodopsin involving the M intermediate were monitored by Ormos et al. (*I161*). Tryptophan perturbation in the L intermediate of bacteriorhodopsin was investigated by Maeda et al. (*I162*). Cladera et al. (*I163*) analyzed the secondary structure of bacteriorhodopsin. The secondary structure of bacteriorhodopsin in solution was investigated by Torres and Padros (*I164*). The photoinduced reaction cycle of bacteriorhodopsin was studied by Deller et al. (*I165*) using picosecond IR spectroscopy. Maeda et al. (*I166*) examined the Schiff base mode of *all-trans*-bacteriorhodopsin and the K and L photointermediates. Takei et al. (*I167*) performed low-temperature studies of Schiff base reprotonation during the M to bR backphotoreaction of bacteriorhodopsin. The proton acceptor of Schiff base deprotonation in bacteriorhodopsin was identified by Fahmy et al. (*I168*). Conformational changes in the M1 and M2 substates of bacteriorhodopsin were characterized by Perkins et al. (*I169*).

Fahmy et al. (*I170*) investigated the structures of bacteriorhodopsin and some of its photoproducts. Time-resolved infrared spectroscopy of bacteriorhodopsin was discussed by Noelker and Siebert (*I171*). Water structural changes in the bacteriorhodopsin photocycle were monitored by Maeda et al. (*I172*). The function of aspartates in proton pumping by bacteriorhodopsin was studied by Fahmy et al. (*I173, I174*). The secondary structure of heat-denatured bacteriorhodopsin was discussed by Cladera and Padros (*I175*). Torres and Padros (*I176*) studied the secondary conformation of bacteriorhodopsin inserted into lipid bilayer membranes. The photocycle of the bacteriorhodopsin mutant Tyr-185 Phe was investigated by He et al. (*I177*). The participation of bacteriorhodopsin active-site lysine backbone in retinal photochemistry was studied by Gat et al. (*I178*).

Ding (*I179*) prepared a thesis which discussed the retinal conformation in rhodopsin and intermediates. High-resolution IR spectra of retinal rhodopsin were acquired by Garcia-Quintana et al. (*I180*). Conformational changes during the rhodopsin/metarhodopsin II transition were investigated by Ganter et al. (*I181*). Structural comparisons of metarhodopsins were made by Klinger and Braiman (*I182*). Masuda et al. (*I183*) observed IR spectra of octopus rhodopsin and its intermediates. The structure of rhodopsin in disk membrane was quantitatively characterized by Garcia-Quintana et al. (*I184*). Walter and Braiman (*I185*) investigated the structure of halorhodopsin in the presence of different anions. The structure of hypsorhodopsin was studied at low temperature by Sasaki et al. (*I186*).

Buchanan et al. (*I187*) monitored light-induced charge separation in *Rhodopseudomonas viridis* reaction centers. Photooxidation of hemes in the cytochrome subunit of *R. viridis* reaction centers was investigated by Nabedryk et al. (*I188*). The secondary quinone environment in *R. viridis* reaction centers was observed by Breton et al. (*I189*). Leonhard and Maentele (*I190*) characterized the vibrational modes of the primary electron donors of *Rhodobacter sphaeroides* and *R. viridis*. The structure of arrestin from bovine retina rod cells was investigated by Garcia-Quintana et al. (*I191*).

The application of vibrational spectroscopy to the study of chlorophylls was presented by Lutz and Maentele (*I192*). Photosynthetic bacteria were studied in vivo by Ozaki and Okada (*I193*). MacDonald and Barry (I194) investigated a novel biochemical preparation of photosystem II. Structural changes of the chromophore of phytochrome were analyzed by Sakai et al. (*I195*). Infrared studies of the triplet state of P680 in the photosystem II reaction center within a chlorophyll dimer were performed by Noguchi et al. (*I196*). The aggregation of bacteriochlorophyll *c* from *Chlorobium limicola* was investigated by Uehara et al. (*I197*). The carbonyl stretching region of the IR spectrum of bacteriochlorophyll-*a* special pair radical cation was studied by Morita et al. (*I198*). The temperature dependence of IR difference spectra of chromatophores and reaction centers from *R. sphaeroides* was investigated by Morita et al. (*I199*).

Time-resolved studies of electron transfer in bacterial photosynthetic reaction centers were performed by Hienerwadel et al. (*I200*). Protein secondary structures in bacterial photosynthetic reaction centers were investigated by Nabedryk et al. (*I201*). Light-induced polarized IR spectroscopy of bacterial photosynthetic reaction centers was studied by Thibodeau et al. (*I202*). Methaquinone photoreduction in bacterial photosynthetic reaction centers was monitored by Breton et al. (*I203*). The temperature dependence of the IR spectrum of bacterial photosynthetic reaction centers was reported by Morita et al. (*I204*). Leonhard et al. (*I205*) characterized primary electron donor "P" oxidation in bacterial photosynthetic reaction centers. Spectra of electrochemically and photochemically generated quinone anions in bacterial photosynthetic reaction centers were studied by Bauscher et al. (*I206*). Morita et al. (*I207*) measured the temperature dependence of IR difference spectra of chromatophores and reaction centers from photosynthetic bacteria.

The secondary structure of glycoproteins of human HIV type 1 was analyzed by Decroly et al. (*I208*). Neoplastic cells in normal and leukemic lymphocytes were characterized by infrared spectroscopy in studies by Benedetti et al. (*I209*). The structure of botulinum neurotoxin type A was investigated by Singh et al. (*I210*). An in vivo study of the state of order of the membranes of Gram-negative bacteria was performed by Schultz and Naumann (*I211*). The characterization of microorganisms by IR spectroscopy was discussed by Naumann et al. (*I212*). Oxygenation of skeletal muscle was monitored by De Blasi et al. (*I213*) using near-IR spectroscopy. Early mineral deposits in bone and enamel were characterized by Rey et al. (*I214*). A study of the carbonate ions in bone mineral during aging was performed by Rey et al. (*I215*). Pleshko et al. (*I216*) studied the interaction of poly(methyl methacrylate) with the protein and mineral contents of bone.

Wong et al. (*I217*) studied phosphodiester stretching bands in IR spectra of human tissues and cultured cells. Infrared microspectroscopy was used to detect the presence of silicone in human breast tissues by Centeno and Johnson (*I218*). Hopkins et al. (*I219*) probed the feasibility of correlating the amide I to amide II absorbance ratio in human hair with characteristics such as age, sex of source, and color. Urinary calculi were analyzed by IR spectroscopy in experiments by Krausova et al. (*I220*). Secondary renal calculi were studied by Ahmed and Khalid (*I221*).

Applications of pressure-tuning IR spectroscopy to cancer research and diagnosis were discussed by Wong (*I222*, *I223*) and Wong and Rigas (*I224*). Prestrelski and Arakawa (*I225*) studied the solution structure and conformational dynamics of tumor necrosis factor alpha. Wong et al. (*I226*) observed distinct structural features in liver tumor tissue of mice. Infrared spectra characteristic of malignant colon tissues were discussed by Rigas and Wong (*I227*). Evidence for extensive structural changes during carcenogenesis in exfoliated human cervical cells was presented by Wong et al. (*I228*). Infrared spectra of human basal cell carcinoma were discussed by Wong et al. (*I229*).

Structural details of the peptide amyloid characteristic of type II diabetes were discussed by Ashburn et al. (*I230*). Studies of the pairing of the separated A and B chains of insulin and its derivatives was studied by Wei et al. (*I231*). An apparatus for the ex vivo measurement of blood sugar levels was described by Clift (*I232*). Multivariate calibration was used to determine glucose in whole blood in experiments by Haaland et al. (*I233*) and by Ward et al. (*I234*). Handheld instruments for the noninvasive measurement of blood glucose were discussed by Rosenthal (*I235–237*), Norris (*I238*), Robinson et al. (*I239*) and by Harjunmaa et al. (*I240*).

Changes in human fetal cerebral hemoglobin concentration during labor were measured by Peebles et al. (*I241*). Stroke-induced changes in lipids and proteins of gerbil brains were monitored by Carney et al. (*I242*). Thorniley et al. (*I243*) discussed the noninvasive monitoring of cerebral tissue oxygenation. Changes in cerebral oxygenation during chemically induced seizures in rats were monitored by Hoshi and Tamura (*I244*, *I245*). Cerebral oxygen saturation as a function of age, sex, and skin color was studied by Dujovny et al. (*I246*) using near-IR spectroscopy. An evaluation of the metabolic recovery after hemorrhagic shock in rats using near-IR spectroscopy was performed by Tamura (*I247*). Near-IR imaging of atheromas in living arterial tissue was presented by Cassis and Lodder (*I248*). Fabian et al. (*I249*) compared human and Dutch-type Alzheimer β-amyloid peptides using IR and circular dichroism.

Changes of some mononucleotide structures induced by drug interactions were measured by Hernanz and Navarro (*I250*). Infrared linear dichroism was used to study nucleic acid/drug interactions by Fritzsche and Rupprecht (*I251*). Coordination chemistry of vitamin C was studied by Tajmir-Riahi and Boghai (*I252*). Cadrin et al. (*I253*) observed alteration in the molecular structure of cytoskeleton proteins in mouse liver treated with griseofulvin using pressure-tuning IR spectroscopy. Salmain et al. (*I254*, *I255*) developed a method for the quantitative trace analysis of transition metal carbonyl-labeled ligands of estradiols and phenobarbital. Villaverde and Morros (*I256*) studied the molecular interaction between the local anesthetic tetracaine and phospholipidic membranes.

The potential of near-IR to study the water content and structure of animal tissues was demonstrated by Ozaki et al. (*I257*). Infrared spectroscopy was used to differentiate between agar- and carageenan-type seaweed galactans in studies by Matsuhiro and Rivas (*I258*). Changes in chemical composition of the tobacco plant during ripening were observed by Eross-Kiss et al. (*I259*). Spectra of raw and modified pineapple leaf fiber were discussed by Saha et al. (*I260*). Sowa et al. (*I261*) monitored structural changes in lipids and proteins in pollen grains. Microbial degradation of plant cell walls was characterized by Delort-Laval et al. (*I262*). Structural studies of cucumber mosaic virus were conducted by Piazzolla et al. (*I263*).

A dissertation by Garcia Lagombra (*I264*) discussed the use of near-IR spectroscopy for the nutritional analysis of feedstuffs. A procedure for the rapid determination of protein in millet was described by Shi et al. (*I265*). A method to measure metabolizable energy in complete poultry feeds was presented by Valdes and Leeson (*I266*). A study of interactions between phytate and protein in rapeseed was performed by Klepacka (*I267*). The effect of solvents on the structural properties of soybean proteins was investigated by Moharram et al. (*I268*). Conformation of wheat gluten proteins in solution and doughy states was discussed by Pezolet et al. (*I269*). A spectral method for the quantitation of mold in barley was presented by Roberts et al. (*I270*). The potential of IR spectroscopy to measure fungal contamination in corn was investigated by Greene et al. (*I271*). Near-IR reflectance spectroscopy was used to measure dry matter disappearance and cell-wall concentration of flaccidgrass masticates in studies by Villalobos et al. (*I272*). Magnesium concentration in perennial ryegrass was measured by Smith et al. (*I273*). The role of disulfide bridges and phospholipids in the stabilization of the α-helix structure of a wheat phospholipid transfer protein was discussed by Desormeaux et al. (*I274*). Chamel and Marechal (*I275*) characterized isolated plant cuticles. Hauber et al. (*I276*) correlated isoenzyme variation in *Phragmites australis* with IR reflectance spectra.

(J) SURFACE TECHNIQUES AND APPLICATIONS

Single Crystals and Other Low Surface Area Materials. Reflection absorption IR spectroscopy (RAIRS) was used by

Hoffmann et al. to study the adsorption and decomposition of ethyl iodide on a Pt(111) surface (*J1*). The molecule adsorbs molecularly at 100 K, lying down initially and standing up as coverage increases. Ethyl, ethylene, and ethylidyne were identified as decomposition products. Kuhn and co-workers have used RAIRS of adsorbed CO to study bimetallic surfaces prepared by evaporation of low coverages of one metal on the single crystal surface of another metal (*J2*). Fan and Trenary have used RAIRS to study the coadsorption of PF_3 and CO, NH_3, or Xe on Pt(111) (*J3*). Similarly, the coadsorption of CO and hydrogen on Ni(110) and CO with Xe on Ni surfaces was investigated using RAIRS by Haq et al. (*J4*) and Xu et al. (*J5*), respectively.

The infrared line shape of molecules adsorbed on a metal surface was described by Mal'shukov, and the results were used to interpret the vibrational spectra of Co/Cu(100) and H/W(100) (*J6*). The influence of the metal surface is said to produce strongly asymmetric bands for dipoles oriented parallel to the surface and weakly asymmetric bands for vibrations along the surface normal. For a CO molecule adsorbed on a metal surface, Volpilhac et al. described how the linewidth of the C–O stretching mode can be related to strength of the metal–CO bond (*J7*).

The use of a tunable diode laser for RAIRS studies was demonstrated by White et al. using a study of the adsorption of CO on Pt(111) (*J8*). The ability to accurately measure the line shapes of absorption peaks was said to offer the greatest potential of this technique. Beckerle et al. investigated the vibrational dynamics of CO adsorbed on Pt(111) using a subpicosecond IR pump/probe method to measure the line shape (*J9*). After the initial strong pump pulse (0.7 ps duration) from a laser tuned to the 2106-cm^{-1} resonance of on-top CO, a weak laser pulse was used to measure the reflection absorption spectrum at varying delay time, CO coverage, surface temperature, and fluence of the pump laser. Yodh and Tom developed a phase-sensitive polarization modulation technique using a picosecond IR source operating at 76 MHz to enable them to measure the 1% changes in reflection with a signal/noise ratio of 10 in less than 1 min (*J10*).

An example of a catalytic reaction studied with IR was the study of CO oxidation on Pd, Rh, and Pt foils published by Coulston and Haller (*J11*). These authors used an FT spectrometer to measure the rotationally resolved IR chemiluminescence of desorbing product CO_2. They found that the product in all the reactions was vibrationally excited. Both RAIRS and transmission IR absorption spectroscopy were used by Yoshinobu and co-workers to explore the effect of UV photoirradiation on chemisorbed CO species on NiO which was supported on either a Ni(111) surface or a porous SiO_2 catalyst (*J12*). The CO was found to photodesorb with different cross sections, depending on the adsorption site.

Carbon monoxide and carbon dioxide adsorption have both been variously used to characterize the surfaces of metals and catalytically active materials by probing their adsorption state with IR spectroscopy. The characterization of oxide surfaces by IR of adsorbed CO was investigated by Pacchioni et al. (*J13*) using an ab initio cluster model calculation to describe the interaction at surfaces of magnesia and nickel(II) oxide. Bolis and colleagues have used a Temkin-type model to describe the adsorption of CO on group IV transition metal dioxides (*J14*). The use of CO_2 for probing certain Lewis acid surface sites was discussed by Morterra et al. (*J15*). These authors described several families of chemisorbed CO_2 species that can be detected in various cases, including end-on σ-coordinated CO_2 that occurs at Lewis acid sites. Heidberg and co-workers have used polarization modulation FT-IR to study the adsorption and 2D gas/solid phase transition of CO_2 on a NaCl(100) surface (*J16*). Berg et al. (*J17*) have explained the temperature dependence of the IR spectra of CO_2 on NaCl(100) found in ref J12 in terms of tilting of the molecule away from the surface and the effects of domain size and multiple layers, using computational and analytical models. Heidberg and Meine have studied the polarized IR spectra of CO_2 adsorbed on MgO(100) single crystal surfaces (*J18*). Heidberg and co-workers (*J19*) have also studied the adsorption of CO on NaCl(100) at low temperatures (>19 K) and Poppe et al. have provided a theoretical basis for the interactions of CO on this surface (*J20*).

The examination of polymer surfaces with IR spectroscopy is a well established technique to probe changes caused by surface modification. Brack and Risen have used specular reflectance IR to characterize the surfaces of chromic acid etched surfaces of high and low density polyethylene (*J21*). Shi et al. used FT-IR combined with X-ray photoelectron spectroscopy (XPS) and contact angle measurements to characterize the results of Ar and O radio frequency plasma functionalization of the model polymer surfaces of hexatriacontane and stearyl stearate films (*J22*). Polysulfone membranes were characterized by Fontyn and co-workers using ATR/FT-IR fast atom mass spectrometry and XPS (*J23*) before and after fouling. The interaction between poly(methyl methacrylate) (PMMA) and oxidized aluminum was investigated by Sondag and Raas (*J24*) using FT-IR multiple specular reflectance (MSR) and inelastic tunneling spectroscopy. Ultrathin (ca. 1 nm) films of the polymer on the oxidized Al surface were scanned with FT-IR MSR in an attempt to probe through the PMMA to the interface. The use of ATR/FT-IR for surface profiling of polymers is the subject of a paper by Chen and Fina (*J25*). The authors modeled the ATR intensities expected from oriented polymer systems with a Laplace transform of the absorption coeffients and compared the latter with the exact intensity calculation using anisotropic Fresnel coefficients.

Thin Films, Langmuir–Blodgett, and Self-Assembled. Both IR absorption and Raman spectroscopy were used by Nakamura et al. (*J26*) to characterize hydrogenated amorphous carbon (a-C:H) films formed by plasma decomposition of toluene. A weak band at 3304 cm^{-1}, attributed to CC–H triple bonds disappeared after annealing the films at 200 °C. MacMillan et al. used IR reflectance to characterize thin aluminum nitride films made by chemical vapor deposition of organometallic compounds on various substrates (*J27*). The authors compared the measured spectra to spectra calculated by using a Lorentz oscillator based model utilizing parameters from the literature. Ohta et al. applied FT-IR reflectance to measure the optical phonons of 0.25–2.0 μm thick films of FeF_2 grown epitaxially on ZnF_2 (*J28*). Spectral changes which occurred as a function of film thickness were adequately fit using a simple oscillator model. They attributed a band observed at 115 cm^{-1} to a surface phonon. Muroya and Uchida combined FT-IR reflection/absorption with X-ray photoelectron spectroscopy to characterize what they called filmy oxides of group III (Al_2O_3) and group IV (SiO_2, GeO_2, SnO_2, PbO_2, TiO_2, ZrO_2) and transition metal oxides formed on metal surfaces (*J29*). The films were prepared by dip-coating the substrate using colloidal and metal resinate solutions. The authors found a linear correlation between the O(1s) binding energy from XPS and the frequency of the M–O vibration detected in IR. Tochigi et al. used polarization modulation combined with FT-IR to allow them to measure IR emission spectra of thin films of lubricants on metal surfaces (*J30*). The polarization modulation eliminated the large background radiation that would otherwise prevent these sorts of measurements. The authors applied the method to characterize films of perfluoroalkyl ether and poly(vinyl acetate).

XPS and IR spectroscopy in the multiple internal reflection mode were used by Niwano et al. to study the effect of UV ozone cleaning of Si surfaces (*J31*). Garrido et al. followed the effect of various Si wafer cleaning processes with reflection absorption IR (*J32*). These authors were also able to use the IR spectra to determine stresses formed as a function of oxidation, processing, and annealing. Mauckner et al. combined FT-IR with time-resolved photoluminescence to study porous silicon in situ during etching, in air under laser exposure, and after chemical oxidation (*J33*). The sensitivity of IR external reflection spectroscopy can be very low on nonreflective surfaces. Nishikawa and co-workers found that they could improve the spectral sensitivity by evaporating thin silver films on the surfaces being analyzed or casting organic films on metallic surfaces (*J34*). The higher sensitivity was attributed to an enhancement of the incident IR radiation field at the metal surfaces. The effect was demonstrated for BaF_2, Ge, glass microscope slides, and poly(acrylonitrile–butadiene–styrene) resin, and the experimental and theoretical aspects to optimizing the optics were discussed. Bermudez also has addressed the issue of enhancing the surface sensitivity of external reflection IR for low-reflectivity substrates (*J35*). He modeled mid-IR reflection from a nonabsorbing layer of varying thickness on a metallic substrate based on Fresnel's relations. The reflection absorption intensity was then calculated for an ultrathin layer of an IR-absorbing molecule on top of the nonabsorbing layer. In a separate paper Bermudez presented the results of a study of fluorine absorption from xenon difluoride and oxygen coadsorption on silicon in ultrahigh vacuum and under low-pressure steady-state conditions with IR reflection absorption spectroscopy (*J36*). IR reflectivity can also be used for determining the charge carrier concentration and mobility in semiconductors. For instance, Mezerreg et al. have fit their experimental IR spectral data to a nonlinear computer model to determine the carrier concentration and mobility and the carrier scattering in n- and p-type gallium antimonide (*J37*). Their results from the reflectivity measurements compared favorably to those determined from Hall measurements.

IR reflection (both internal and external) absorption and transmission spectroscopy have all played key roles in the study of Langmuir–Blodgett (L–B) and self-assembled monolayer films. These techniques by themselves and especially used in combination provide information on chemical interaction with the substrate and the structural and orientational aspects of thin films. Mielczarski pointed out that optical effects which vary with the substrate contribute to the spectral features observed in reflection absorption IR spectroscopy (*J38*). To allow one to relate spectral differences to structural or chemical bonding differences in thin films, the author maintains it is crucial to distinguish the effects of the substrate. The electrical field present within an organic monolayer was examined under various optical conditions to determine the effects on the spectra of multilayer systems. Self-assembled monolayers of cuprous ethyl xanthate films on copper, cuprous sulfide, and water were examined. Buontempo and Rice discussed the theory of IR external reflection spectroscopy and applied the technique to determining the orientation of adsorbates on dielectric substrates without the concomitant use of other techniques (*J39*). Parikh and Allara developed a semitheoretical formalism that used classical electromagnetic theory to determine quantitative molecular structure in L–B films (*J40*). Their formalism can be applied to both external and internal reflection and transmission spectroscopies as well as X-ray reflectivity and UV/visible ellipsometry. The approach was tested by simulating spectra of specific self-assembled and L–B film systems. The resulting spectra compared favorably in cases in which independent results were available. Ahn and Franses applied ATR/FT-IR dichroic ratio methods to determine the orientation of chain axes in L–B films of calcium, cadmium, and lead stearate on Ge, hydrophobic and hydrophylic Si plates (*J41*).

In addition to characterizing L–B and self-assembled films, reflection IR is also useful for monitoring changes in prepared films caused by chemical reaction or physical processes. Buontempo and Rice applied IR external reflection spectroscopy to follow phase transitions in L–B films of heneicosanol (*J42*). These authors monitored the CH_2 symmetric and antisymmetric stretching modes as a function of surface pressure and temperature. Yamamoto and co-workers used IR reflection absorption to monitor the photodimerization of stillbazolium cations incorporated into L–B films (*J43*). The esterification and amidization of alkanethiol monolayers on gold were studied by Duevel and Corn using polarization modulation FT-IR (*J44*). Yano et al. studied the electron beam induced reactions in stearic acid L–B films with multiple internal reflection IR spectroscopy (*J45*).

Tribochemical and thermochemical reactions of stearic acid on copper surfaces were studied with IR microspectroscopy by Hu et al. (*J46*). The tribochemistry was initiated using a pin on disk device under boundary lubrication conditions. The authors were able to identify copper stearate complexes that supposedly formed a protective film on the surfaces. The characterization of the structure of (3-aminopropyl)triethoxysilane films on silicon oxide was studied with IR reflection spectroscopy, scanning electron microscopy, atomic force microscopy, ellipsometry, and XPS by Vandenberg and colleagues (*J47*). The films were prepared under various conditions of temperature and time using different solvents and cured in various manners.

The orientation of water-soluble surfactants at the air/water interface was explored by Tung et al. using IR reflection absorption spectroscopy (*J48*). Gericke et al. used the same technique to determine the structure of hexadecan-1-ol at the water/air interface (*J49*). The latter authors varied the angle of incidence and polarization and compared their experimental results to theory.

The use of in situ FT-IR to study surfaces important for electrochemical reasons has continued to be a dynamic field over the last two years. The adsorption of methanol on Pt electrodes in acid medium was monitored using electrode potential modulated IR spectroscopy (EMIRS) by Lopes et al. (*J50*). The authors obtained spectra at frequencies below 1050 cm^{-1}, a spectral region not available to them before. Xing et al. used in situ FT-IR reflection absorption spectroscopy to monitor the electrooxidation of L-ascorbic acid on Pt electrodes in acid media (*J51*). The migration of adsorbed CO between various surface sites on Pt(100) and -(111) single crystal electrode surfaces was studied by Nakamura et al. using EMIRS and time-resolved reflection absorption IR spectroscopy (*J52*). The adsorbed CO interconverted between bridge bonded and on-top sites according to electrode potential. Chang et al. used IR spectrophotometry to determine the coverage of CO on Rh(100) electrodes and compared the results to simultaneous voltometric measurements (*J53*). Substantial errors were found in the latter technique and were discussed in terms of Faradaic charge arising from the oxidation of the surface. Popenoe described the design considerations and testing of a new cell constructed to allow the in situ measurement of IR reflection spectra of monolayer films of long-chain alkanethiolates on evaporated Au films beneath a thin layer of aqueous solution without the need for electrochemical or polarization modulation (*J54*).

Catalysts and Other High Surface Area Materials. IR spectroscopy has played a significant role in the characterization of catalyst and elucidation of catalytic pathways through the study of catalytic kinetics and the identification of intermediate species. The acidity of alumina-supported catalysts was probed by Wachs' group using IR spectroscopy to monitor the amount and type of hydroxyl groups present on the surface (*J55*). Similarly, Topsoe and Topsoe have used IR spectroscopy to monitor the acidity of Mo/alumina catalysts (*J56*). Lewis acid Al^{3+} sites on the dehydroxylated surface of Al_2O_3 were monitored by Ballinger and Yates using IR spectroscopy of CO adsorbed at low temperature (*J57*). Booker and Keiser used IR to monitor the titration of OH groups with Rh^{3+} (*J58*). The OH bands at 3682 and 3741 cm^{-1} decreased with increasing Rh^{3+} coverage, without affecting other OH bands. Echoufi and Gelin used IR spectroscopy of adsorbed CO at low temperature to characterize decationated y-type zeolites (*J59*). Kappers and Van Der Maas found IR spectra of adsorbed CO useful for characterizing the coordination number of Pt in Pt catalysts made with different supports (*J60*). The frequency of the C–O stretch of linearly bonded CO varied at face, edge, and corner atoms. Bagshaw and Cooney used IR spectroscopy of adsorbed pyridine as a probe of the acidity of alumina, zirconia, and titania pillared clays (*J61*). IR evidence for the formation of tilted carbon monoxide on an alumina-supported Ni catalyst was reported by Ma et al. (*J62*).

The large background photoemission of activated H-erionites was investigated by diffuse reflectance FT-IR spectroscopy (DRIFT) and Raman spectroscopy by Salzer and co-workers (*J63*). The activated zeolites were sealed in glass tubes for the DRIFT spectroscopy.

Lapinski and Ekerdt described a method for obtaining kinetic parameters for surface hydrocarbon reactions using FT-IR (*J64*). They applied their method to the study of the reaction of ethylene on alumina-supported nickel. Ballinger and Yates used transmission IR spectroscopy to monitor the catalytic decomposition of 1,1,1-trichloroethane on high surface area alumina (*J65*). The strong metal support interaction in dilute Pt/alumina catalysts was studied by Abasov and colleagues with DRIFT and adsorption of H_2 and CO (*J66*).

IR spectroscopy is particularly useful for identifying intermediates in catalytic reactions. The identification of intermediates in hydrocarbon reactions has been pursued by several groups. Szilagyi reported the presence of ethylidyne after the adsorption of ethylene on a silica-supported Pt catalyst (*J67*). At large exposures of ethylene, Szilagyi identified vinyl groups formed on the surface. In contrast De la Cruz and Sheppard identified an adsorbed ethyl group on the surface after adsorption of ethylene on a Pt/SiO_2 catalyst (*J68*). Datka and Eischens used IR spectroscopy to study the decomposition of acetylene on a Pt–Sn/alumina catalyst suspended from a Cahn microbalance (*J69*). The latter authors followed the deposition of coke on this catalyst and determined that the coke contained a carboxylate species.

An in situ high-pressure (503 K, 10 MPa) FT-IR study of CO_2/hydrogen interactions on zinc oxide/SiO_2, Cu/SiO_2, and Cu/zinc oxide/SiO_2 methanol synthesis catalysts was made by Millar and co-workers (*J70*). Formate species were detected as intermediates which were eventually hydrogenated to methanol. Bijsterbosch and co-workers have described a high-vacuum cell for high-temperature in situ infrared studies of heterogeneous catalysts (*J71*). A particularly important aspect to their cell design is its ability to control the gas phase exposed to the catalyst.

(K) POLYMER APPLICATIONS

Numerous investigators have used infrared spectroscopy for polymer characterization. A summary of applications for much of 1991 and 1992 may be found in a review article by Smith et al. (*K1*).

Tikuisis et al. (*K2*) quantitatively determined starch in polyethylene. Yan et al. (*K3*) measured the effect of radiation-induced cross-linking on tie molecule relaxation during annealing of oriented low-density polyethylene. Miller (*K4*) used near-IR spectroscopy in the composition determination of high-density/low-density polyethylene blend films. Chen and Fina (*K5*) described a method for obtaining orientation gradients on isotactic polypropylene films. Bruce et al. (*K6*) studied molecular orientation in uniaxially compressed isotactic polypropylene disks. Nishioka et al. (*K7*) studied the slip-reducing mechanism in polypropylene-laminated films. Polyethylene/nylon laminates were analyzed nondestructively in experiments by Miller et al. (*K8*). Interactions between ethylene–(vinyl acetate) copolymer and polyethylene were investigated by Ray et al. (*K9*). Interfacial interactions between hydrated aluminum and polar groups in ethylene copolymers were examined by Straalin and Hjertberg (*K10*).

Yang (*K11*) analyzed the interface between polyolefins and trialkylamine primer. The kinetics of rubber surface oxidation of EPDM-40 rubber was modeled by Kondyurin (*K12*). The effect of temperature and time on the thermal degradation of polybutadiene was discussed by Schneider et al. (*K13*).

Quantitative analysis of acrylic copolymers was described by Mao et al. (*K14*). Kinetic studies of photopolymerization of cyanoacrylates were reported by Yang (*K15*). Infrared and Raman studies of methyl methacrylate and butadiene were performed by Edwards et al. (*K16*). The interactions between surfactant and copolymer in latex films were investigated by Thorstenson and Urban (*K17*, *K18*) and by Timothy et al. (*K19*). Hydroxyl number in acrylics and polyesters was determined by Lee et al. (*K20*). The use of reactive rubbers as toughening agents for polyesters was discussed by Martuscelli et al. (*K21*). Pyrolysis gas chromatography/infrared spectroscopy of aromatic polyesters and copolyesters was presented by Hummel et al. (*K22*). Dipole/dipole interactions in esters and liquid crystal polyesters were studied by Galbiati et al. (*K23*). The effect of the OH group of phenoxy resins on miscibility with other polymers was discussed by Fernandez-Berridi et al. (*K24*). Stopped-flow studies of the kinetics of polycarbonate polymerization were presented by Brittain et al. (*K25*).

Ion beam induced transformations in polyimide films were characterized by Xu et al. (*K26*). Proton conduction in poly(acrylamide)–acid blends was monitored by Rodriguez et al. (*K27*). A description of the urethane group association scheme in poly(ether urethane) elastomers was presented by Zharkov et al. (*K28*). DeHaseth et al. (*K29*) characterized polyurethane foams using mid-IR-transmitting optical fibers.

Cowie et al. (*K30*) studied surface enrichment in polystyrene(poly(vinyl methyl ether)) blends using attenuated total reflectance (ATR) spectroscopy. Structural changes in acrylonitrile–butadiene–styrene copolymer thin films after iodine doping were examined by Bakr (*K31*). Sargent et al. (*K32*) studied the photooxidation of styrene–acrylonitrile copolymers. The desorption of emulsifier from the polystyrene particle surfaces of latexes was followed by Stone-Masui and Stone (*K33*). The influence of solvent on the electrophoretic mobility of polystyrene latex was investigated by Verdegan and Anderson (*K34*). Miscibility of blends of poly(vinyl methyl ether) and styrene containing polymers was monitored by Ryou et al. (*K35*) and Ha et al. (*K36*).

Voice et al. (*K37*) studied molecular orientation in poly(aryl ether ether ketone) (PEEK). Cole and Casella (*K38*) studied the thermal degradation of PEEK–carbon composites. The cyclic anhydride intermediate involved in the ester crosslinking of cotton cellulose was identified by Yang (*K39*). Model epoxy networks were characterized by Gallouedec et al. (*K40*). Cure kinetics of liquid crystalline epoxy resins were investigated by Carfagna et al. (*K41*). Buechtemann et al. (*K42*) examined the IR spectra of poly(vinylidene fluoride) films exposed to cyclic electric fields. Besbes et al. (*K43*) studied segmental orientation in dry and swollen poly(dimethylsiloxane) networks. Watanabe et al. (*K44*) studied heat-treated poly(*n*-propylsilyne) with far-IR spectroscopy. Orientation in poly(arylenevinylene) copolymers and blends was measured by Gregorius and Karasz (*K45*).

(L) TECHNIQUES AND APPLICATIONS OF IR REFLECTION SPECTROSCOPY

Ohta and Ishida described a novel iterative method for determining optical constants from ATR/FT-IR spectra and applied it to several polymer films cast directly on the internal reflection element (IRE) from solution with results comparable to methods using transmission spectra (*L1*). Marley et al. compared cylindrical internal reflectance spectroscopy and transmission spectroscopy of liquids to develop a simple calibration procedure to predict effective path length and calculate sample concentrations and IR absorption coefficients for aqueous solutions (*L2*). Bertie et al. evaluated the accuracy of current computational methods for determining optical constants from multiple ATR measurements using the CIRCLE cell (*L3*). Stuchebryukov and Rudoi investigated the lack of ideal contact between samples with rough surfaces and IREs and proposed methods to circumvent this problem and allow quantitative ATR measurements (*L4*). Afran discussed the advantages of square column ATR compared to cylindrical cells for the quantitative analysis of aqueous solutions, including high-absorbance linearity (*L5*). Gee and Hicks reported a study of adsorption of hydrogen atoms on gallium arsenide by multiple internal reflection IR spectroscopy (*L6*). Pedraza et al. deposited thin films of 316L stainless steel on germanium cylindrical IREs to study the stability of the samples in aqueous solutions. They reported that the deposition of chromium oxide on the substrate prior to the deposition of the steel improved the film adhesion and the optical properties of the substrate (*L7*). Lynch et al. reported that overlaying inorganic and organic solid powder samples with nondissolving liquids enhanced spectra obtained by ATR/FT-IR (*L8*). Huang and Urban developed and evaluated a new ATR algorithm using Kramers–Kronig (KK) transforms (*L9*). Tickanen et al. developed a method for using variable-angle ATR/FT-IR and the subtractive form of the KK transform to determine optical constants for opaque samples (*L10*). Huang and Urban presented a new linear interpolation approach to quantitative depth profiling of surfaces using ATR/FT-IR (*L11*). Johnson and Doblhofer investigated the use of barium fluoride as an IRE for ATR spectroelectochemistry by sputtering gold or platinum electrodes onto a hemispherical element (*L12*). Johnson et al. discussed the application of ATR/IR for electrochemical studies (*L13*). Chiacchiera and Kosower reported the use of silver halide fibers for ATR measurements of microsamples (*L14*). Durrer et al. used ATR/FT-IR to detect latexes at the surface of excised intestinal mucosa of rats and studied the adsorption kinetics of mucoadhesive latexes (*L15*).

Miser et al. applied ATR/IR to measure diffusion coefficients of plasticizers in nitrocellulose, cellulose acetate butyrate, and their blends (*L16*). Yuan and Sung developed a rotatable truncated hemispheric ATR attachment and used it to obtain horizontal angular absorbance profiles from polymer surfaces (*L17*). Jensen et al. used ATR/IR to develop penetration profiles in wood treated with resins (*L18*). Rosenberg and Kellner used ATR/IR to study diffusion of glucose into a PVA membrane (*L19*). Fieldson and Barbari applied ATR/IR to measure the diffusion of water into polyacrylonitrile (*L20*). ATR/FT-IR was used to investigate the interdiffusion in poly(vinyl methyl ether) and polystyrene by Jabbari and Peppas (*L21*). Huang et al. applied ATR/IR to study surface crystallinity in polyethylene (*L22*). Semwal et al. investigated sulfur mustard desorption through paint films using ATR/IR with data obtained applicable to the selection of appropriate paint films for protection (*L23*). Hong et al. applied ATR/IR to investigate the chemical and physical changes with depth in poly(vinyl fluoride) film caused by flame treatment (*L24*). Dillon and Hughes characterized polyurethane gastric bubbles by ATR/IR after use in vivo for up to four months, with results indicating significant changes in the bubble material over time (*L25*). Jeon et al. used ATR/IR to quantitatively analyze the extent of bovine serum albumin adsorbed onto biomedical grade polyurethane deposited on a ZnSe IRE (*L26*). Van de Voort et al. developed an ATR/IR method to determine the iodine value and saponification number of fats and oils with results comparable to current methods (*L27*). De Haan et al. reported the application of ATR/FT-IR to investigate moisturization of skin using liquid crystalline creams (*L28*). Ling et al. described the use of multiple reflection IR spectroscopy as a nondestructive technique for the study of silicon wafer surfaces (*L29*).

(M) QUANTITATIVE ANALYSIS

Martens et al. (*M1*) reported on a spectroscopically meaningful way of updating multivariate bilinear calibration models. The method, which applies to both one single *Y*-variable and a set of *Y*-variables attempts to span the *X–X* and *X–Y* covariances involved by constructing artificial bilinear samples that can be analyzed together with updating data, was illustrated with an example from near-IR process monitoring. Salgo et al. (*M2*) used near-IR to determine glucosinolates and erucic acid in whole rapeseed. Garcia-Ciudad (*M3*) predicted the protein content of seminatural grasslands taken at various stages of maturity using near-IR spectroscopy. Bye (*M4*) used multivariate calibration by partial least squares regression and IR spectroscopy to analyze mixtures of crystalline and amorphous silica present in dust samples. The procedure was found to be accurate, rapid, and low cost. Karstang and Henriksen (*M5*) used IR spectroscopy along with mutivariate calibration to determine additives present in high-density polyethylene. Lanher (*M6*) reported on a new software package, ANAQUANT, specifically designed to the quantitation of compounds in biological products. The functionality and validity of the software was tested by measuring fat and protein in liquid cow's milk using FT-IR and a hand-crafted transmission flow cell. Nishiyama et al. (*M7*) used near-IR reflectance spectroscopy to determine the concentration of the natural sweetener stevioside in *Stevia rebaudiana* leaves.

Faix and Boettcher (*M8*) determined the phenolic hydroxyl group content in milled wood lignins by FT-IR spectroscopy along with partial least squares or principal component regression analysis. Principle component regression was found to be slightly inferior to partial least squares. Forrest (*M9*) used FT-IR spectroscopy to detect and determine hydroxypropylation of starch. IR spectra of hydroxyproylated starch and its unmodified precursor were acquired. The second derivative of the difference spectrum between the two was calculated and the area of the peak centered at 2974 cm^{-1} compared to that of a calibration curve. Wallbaecks et al. (*M10*) used near-IR, FT-IR, and ^{13}C CP-MAS NMR along with partial least squares and principal component analysis to probe the relationships between different sample properties and to test the predictive efficiency of the models. Seven fully bleached pulp samples originating from the same batch of pine wood but taken from different pulping processes were used in the evalutation.

Morimoto and Nishio (*M11*) determined the octane number of gasoline using FT-IR spectroscopy and principle component analysis or multiple linear regression analysis. Dupuy et al. (*M12*) discussed several sample preparation techniques for the quantitative analysis of food and agroindustrial products using multilinear regression mathmatical methods. Attenuated total reflectance was found to provide the best results for concentrated solutions. Dupuy et al. (*M13*) used attenuated total reflectance FT-IR and partial least squares regression analysis to determine glucose, fructose, and sucrose in powdered sugars.

Ishida (*M14*) discussed the application of optical theory to quantitative surface analysis using FT-IR. Special emphasis was placed on applying optical theory and mathematical algorithms to molecular depth profiling. Wangmaneerat et al. (*M15*) and Haaland (*M16*) determined the phosphorus concentration of thin films of phosphosilicate glass using emission spectroscopy combined with multivariate calibration. Erickson et al. (*M17*) applied multivariate calibration to provide a method for the quantitative analysis of liquid samples. Differing forms of the data including second-derivative and interferogram representation were used in the prediction of sample composition.

Monfre and Brown (*M18*) report developing a new procedure for performing quantitative analysis in the Fourier domain. The procedure involves the use of a linear recursive digital filter for the analysis of raw interferogram data from a FT-IR spectrophotometer. The method permitted the use of spectral information that is generally neglected when conventional quantitative Fourier-domain procedures are used. Simpson (*M19*) reported using FT-IR and multicomponent partial least squares regression calibration to analyze additive packages in commercial motor oil and to monitor the condition of used oil. The procedures were also used as a quality control technique to discriminate between different formulations of a complex resin based mixture and as a way to routinely determine failure analysis for automobile plastics.

(N) INFRARED INSTRUMENTAL ACCESSORIES

A variety of new infrared accessories have been described in the literature or introduced by instrumental vendors. These include a high-vacuum cell for high-temperature in situ IR studies of heterogeneous catalysts (*N1*), a long optical path cell for photochemical kinetics in heterogeneous systems of low concentration (*N2*), a high-temperature IR absorption gas cell (*N3*), a pyrolysis sampling cell (*N4*), a controlled-environment chamber for atmospheric chemistry studies (*N5*), a simple and efficient low-temperature sample cell (*N6*), a nonpressure-dependent IR absorption sample cell (*N7*), high-pressure diamond anvil sample cells (*N8*, *N9*), external reflection cells for powdered samples (*N10*) and fluid samples at elevated temperatures and pressures (*N11*), internal reflection cells for liquids and/or solids (*N12–15*), a gold minigrid cell for FT-IR spectroelectrochemistry studies (*N16*), remote sampling accessories for reflectance, emission, and transmission analysis (*N17*), an emission accessory (*N18*), and a new temperature-compensated multiple-wavelength mini-IR detector for monitoring parts per million to percent concentration in gases and liquids (*N19*).

(O) FT-RAMAN SPECTROSCOPY

Petty describes the self-absorption effect in terms of how it might be expected to effect near-IR Fourier transform Raman spectroscopy. Experiments are detailed which dramatically demonstrate self-absorption in THF. The results of these experiments are applied to some recently published unexplained observations, and it is thought that these observations may be fully explained by the self-absorption phenomenon. Discussion is presented of how to recognize and avoid or minimize self-absorption in quantitative Fourier transform Raman measurements (*O1*). Schrader et al. discuss the optimization of the sampling arrangement in order to increase the number of samples open to study using near-IR FT-Raman spectroscopy and to improve the quality of the spectra generated. This involves taking into consideration the optical properties of the sample, especially the absorption spectrum and thermal emission characteristics, according to Planck's and Kirchhoff's laws. Only a few samples continue to show continuous backgrounds; this is sometimes true even if no background is apparent with visible excitation. The sources of such backgrounds are described, as are means to reduce or eliminate most of them (*O2*). Walder and Smith evaluated three fundamental behaviors of vibrational spectroscopy data manipulation routinely associated with Fourier transform IR spectroscopy for near-IR Fourier transform Raman spectroscopy. Spectral reproducibility, spectral subtraction, and sensitivity were examined relative to the near-IR FT-Raman experiment. Quantitative predictive ability is compared for identical sets of samples containing mixtures of the three xylene isomers. Partial least squares analysis is used to compare predictive ability. IR performance is found to be better than Raman, though the potential for method development using near-IR FT-Raman is shown to be quite promising (*O3*). Deeley et al. compared Fourier transform IR and near-IR Fourier transform Raman spectroscopy for quantitative measurement of polymorphism in cortisone acetate, a synthetic adrenocortical steroid. For cortisone acetate, similar quantitative precision was obtained with both Raman and diffuse reflectance IR measurements. The Raman measurements of cortisone acetate gave a calibration with a standard error of prediction of better than 2.5%. The combination of excellent precision with very convenient measurement of powders makes near-IR FT-Raman spectroscopy a valuable tool for quantitative measurements of polymorphism (*O4*).

DeBakker et al. monitored the thermal cure of a commercial tetraglycidyl-4,4′-diaminodiphenylmethane/diaminodiphenylsulfone epoxy resin by FT-Raman spectroscopy, and the spectral changes were interpreted in terms of the mechanism of cure. The FT-Raman data obtained were free from fluorescence and enabled the changes to be monitored quantitatively. The kinetic data obtained by this method with those obtained by a parallel study using in situ FT-near-IR spectroscopy effectively reflected the kinetics of the curing process. Evidence was also found from the FT-Raman data that suggested the presence of a particular impurity cyclization reaction in the commercial epoxy resin MY721 (*O5*). Clarkson et al. determined that the application of near-IR FT-Raman spectroscopy for the in situ monitoring of the homopolymerization reactions from commercial acrylate monomers was reliable, reproducible, and quantitative. The effect of reaction temperatures and monomer additives could be elucidated from the corresponding change in reaction time. While the method was widely applicable to commercial systems, it lacked universal usage. The main problem encountered was polymer insolubility, which in turn produced erratic, nonreproducible data. Minor cell modification should overcome this problem (*O6*).

Lewis et al. describe a fiber-optic sample cell designed for use in the near-IR region. For Fourier transform Raman spectroscopy, Nd:YAG laser radiation is transmitted to the sample through one fiber bundle, while a second fiber bundle collects and transfers the scattered light to the source compartment of an IR interferometer equipped with a CaF2 beam splitter and an InGaAs detector. Because the two fiber bundles are mounted in the aluminum body of the cell, virtually no optical alignment is required. In addition to its utility in FT-Raman spectroscopy, the cell may be used for obtaining near-IR absorption spectra of liquids and reflectance spectra of solids without changing its configuration (*O7*).

(P) PHOTOACOUSTIC SPECTROSCOPY

Palmer, Ditmar, et al. employed step-scan FT-IR spectroscopy in combination with photoacoustic detection to depth profile laminated polymeric materials, such as ethylene–vinyl acetate copolymer on polypropylene and Kapton on Teflon. Depth profiling can be performed using a combination of the modulation frequency dependence and phase lag of the photoacoustic signal. The phase analysis technique is particularly useful for cleanly separating the signal due to a thin (<5 μm) surface layer from that of the bulk or substrate (*P1–4*).

Yang et al. utilized FT-IR/PAS to study the oxidation and degradation in various textile fabrics. All the IR spectroscopic data demonstrated that FT-IR/PAS was able to differentiate the near surface of a textile sample from its bulk. Therefore, it could be used to determine the distribution of degradation products between the near surfaces of fabrics and their bulk. Materials that were examined include polypropylene, cotton cellulose, and carbon fiber (*P5–10*). McDonald and Urban examined the Nextel glass fiber/polyimide interface by FT-IR/PAS. The exposure of the fiber matrix interface to dry and aqueous environments may lead to chemical changes at the interface (*P11*). Delprat and Gardette examined photooxidized polypropylene samples by FT-IR/PAS and transmission micro-FT-IR analysis of cross-sectioned layers. The oxidation profiles monitored were shown to arise from oxygen diffusion limitation and also from the heterogeneous absorption of light by the TiO_2-pigmented samples. The intensity of the carbonyl absorbance measured by FT-IR/PAS was determined, and the front layer degradation monitored by both techniques had identical values. This rapid and nondestructive technique may thus be applied to quantitatively measure the surface degradation of aged polymers (*P12*).

Ludwig and Urban monitored the diffusion of ethyl acetate vapor from poly(vinylidene fluoride) films via rheophotoacoustic FT-IR spectroscopy. The effect of stress on the mobility of ethyl acetate is emphasized (*P13*). Coppee studied the cross-linking of two structural adhesives using FT-IR/PAS combined with thermal analysis methods, DSC, and dynamic mechanical spectroscopy. FT-IR/PAS was the only way to investigate the cure of the adhesive films with their carrier (*P14*). Allen et al. investigated the chemical and physical characteristics of sheet-molded compounds using FT-IR/PAS and mechanical spectroscopies. FT-IR/PAS facilitated surface analysis for determination of composition, depth profile, and thermal exposure effects. Dynamic mechanical spectroscopy utilized torsion, tension, and flexure modes to evaluate temperature and frequency-dependent viscoelastic properties (*P15*).

Peck et al. employed FT-IR/PAS to study the appearance and diffusion of byproducts dimorpholinylthione (**I**) and zinc stearate (**II**) in vulcanized natural rubber filled with carbon blacks of different particle size or surface structure and activity. The diffusion of **I** is not affected by the size or surface structure and activity of the carbon blacks used, but the diffusion of **II** is affected by the particle size (*P16*). Raveh et al. determined that phase FT-IR/PAS, when applied to the study of hydrogenated amorphous carbon (a-C:H) films deposited in a dual microwave/radiofrequency plasma, can distinguish between surface and bulk chemical structure. The spectra correlate well with physicochemical properties of a-C:H films (*P17*). Gentzis et al. examined coals from western Canada for structural changes occurring during oxidation, carbonization, and hydrogenation by FT-IR/PAS and optical microscopy. IR spectroscopy was sensitive to the molecular structural changes occurring during these processes and provided a chemical basis for interpreting data from chemical analysis and optical microscopy (*P18*).

Baldwin et al. examined the applicability of FT-IR-based PAS for on-site analysis of low concentrations of volatile organics in air. Band intensities are proportional to concentration, and sensitivities were good. Typical limits of detection are a few ppm (*P19*). Carter and Wright evaluated the appropriate sample position in a FT-IR/PAS experiment. The experimental selection of the distance between the sample and the window in a FT-IR/PAS experiment is an important parameter when one is optimizing the interferogram intensity. Experimentally demonstrated were the advantages of selecting a small distance rather than the full boundary layer thickness required to ensure complete conversion of thermal to pressure signal at the sample surface. The minimum volume of the sample chamber is a controlling factor in the optimization of the experiment. The value of minimizing sample chamber volume through controlling sample thickness is shown while the need for care when doing so is pointed out (*P20*).

(Q) DIFFUSE REFLECTANCE SPECTROSCOPY

Dai et al. have developed a cell of simple design which fits in sample cap holders of Spectra-Tech DR accessories. This new diffuse reflectance IR cell enables the routine measurement of hydroxide species in various anhydrous, but extremely water-sensitive inorganic salts (*Q1*).

White describes the instrumental parameters that determine the accuracy of variable-temperature diffuse reflectance Fourier transform IR spectroscopy (VT-DRIFTS) measurements. Methods for removing baseline artifacts resulting from fluctuations in these parameters are also given. Baseline artifacts in VT-DRIFTS spectra can be minimized by measuring reference and sample single-beam spectra at the same temperature. In addition, VT-DRIFTS baseline errors can be removed by subtracting baseline functions from apparent absorbance spectra (*Q2*, *Q3*).

TeVrucht and Griffiths investigated five materials for use as diluent matrixes for diffuse reflectance IR Fourier transform spectroscopy of powdered samples. DRIFT spectra of powdered carbazole dispersed in germanium, silicon, diamond, potassium chloride, and a chalcogenide glass ($Ge_{28}Sb_{12}Se_{60}$) were compared. Three particle size ranges were investigated for five concentrations of carbazole ranging from 0.1 to 10%. The effects of particle size and refractive index of the matrix are discussed (*Q4*). Mandelis et al. present a self-consistent discontinuum theory describing the dependence of the diffuse reflectance and diffuse transmittance of powders on their optical absorption coefficients. The theory is valid for particle sizes that are large compared to the exciting wavelength. It constitutes generalization and evolution of the statistical theory of absolute diffuse reflectance of powders put forth earlier. Practical experimental limits of the validity of the discontinuum theory are also established (*Q5*).

Boroumand et al. propose an adaptation of Kubelka's general model of diffuse reflectance and diffuse transmittance of light to nonideal scattering samples. It is applied to quantitative Fourier transform IR spectrometry of nondiluted surface-derivatized silica powders. Corrections to the measurements are introduced in order to take into account common problems that often prevent a truly quantitative application of the DRIFTS analytical method. Effects on the measured data due to the background absorption of the sample and to the specular reflection on its surface are discussed and corrected for quantitatively. The possible existence of gradients of the optical properties of the powder medium is taken into account by a numerical adaptation of the model to inhomogeneities of the phenomenological absorption and scattering coefficients K and S (*Q6*).

(R) TIME-RESOLVED INFRARED

Masutani et al. developed a novel asynchronous time-resolved FT-IR spectrometer based on a conventional continuous-scan interferometer, which does not require synchronization between the signal for time resolving and that for sampling of the A/D converter. The time-resolved signal processing assembly, which can be attached to any FT-IR, consists of a pulse generator, a pulse delay circuit, a gate circuit, and a low-pass filter (*R1*). Nakanaga et al. described a simple method for measuring time-resolved FT-IR spectra using a pulsed electrical discharge synchronized to the motion of the moving mirror (*R2*). Beckerle et al. applied subpicosecond pulses to measure transient IR spectra of vibrationally excited (acetylacetonatodicarbonyl)rhodium (*R3*). Urano and Hamaguchi (*R4*) and Sugisawa et al. (*R5*) reported the use of microsecond time-resolved IR to investigate the electric field induced reorientation of nematic liquid crystals. Alden et al. presented a theory of femtosecond time-resolved IR applying the theory of nonstationary time-dependent absorption (*R6*). Weiller et al. used pulsed UV laser photolysis and a rapid-scan FT-IR spectrometer to investigate organometallic carbonyl substitution kinetics in liquid xenon (*R7*). Gregoriou et al. reported the application of step-scan interferometry to the determination of two-dimensional IR spectra and discussed experiments involving a polymer film and nematic liquid crystals (*R8*). Toriumi discussed 1D and 2D time-resolved studies of electrical field induced reorientation dynamics of liquid crystals and cautioned that sample preparation is critical to the success of the experiments for anisotropically oriented liquid crystals (*R9*). Noda et al. developed a time-resolved spectrometer coupled with a dynamic perturbation cell to measure perturbation-induced dynamic fluctuations of IR signals required to generate 2D IR spectra (*R10*). Palmer et al. discussed the application of step-scan FT-IR spectroscopy to the characterization of polymer systems (*R11*). Miranda et al. discussed the application of time-resolved IR reflection and transmission spectrometry to the investigation of semiconductor carrier dynamics and reported that the IR probe is sensitive to the spatial profile of the carrier distribution far below the surface (*R12*). Nowotny et al. reported in situ transmission measurements on single zeolite crystals using time-resolved IR (*R13*). Advantages and limitations of the application of time-resolved IR to general kinetic studies in the microsecond and submicrosecond time domain was discussed by Rimai et al. (*R14*). Turner et al. reviewed the application of time-resolved IR spectrometry to the study of excited states of transition metal species (*R15*). Ford et al. applied flash photolysis with time-resolved IR detection to study kinetics of reactive organometallic intermediates. The pump source used was a XeCl excimer laser and lead salt diode lasers were used as probe sources (*R16*). Bakker and Lagendijk reported a time-resolved IR study of the vibrational relaxation after excitation of the C–H vibration for solutions of bromoform and tetrabromoethane, and pure liquid bromoform (*R17*). Noelker et al. applied submicrosecond time-resolved step-scan FT-IR spectrometry to investigate chromophore and protein reactions of bacteriorhodopsin (*R18*). Souvignier and Gerwert reported improving the time resolution of a conventional fast-scan FT-IR spectrometer by using a stroboscopic technique and applied it to study the proton uptake reaction mechanism of bacteriorhodopsin (*R19*). Gerwert also reported the application of time-resolved IR to study the bacteriorhodopsin photocycle (*R20*). Preses et al. reported the development of an IR time-resolved FT emission spectrometer based on an interferometer combined with a CAMAC-based data acquisition system, which operates in the smooth scan mode (*R21*). Rawlins et al. reported the collection of time-resolved IR emission spectra from the fundamental vibration/rotation band of nitric oxide in a shock tube using a specially constructed IR array spectrometer. The method was applied to the determination of nitric oxide in a high-temperature Mach 3 air flow in the PSI shock-tunnel facility (*R22, R23*).

(S) INFRARED MICROSCOPY

Sommer and Katon conducted model experiments on cellulose acetate film in an effort to quantitatively assess the extent of stray light, resulting from diffraction, in a FT-IR microscope system. The effects of stray light were studied under conditions employing different aperturing modes, aperture sizes, and wavelengths of light. Results and consequences of the findings are discussed with respect to the spatial resolution and quantitative integrity of the data obtainable in mapping analyses of multilayer plastic laminates (*S1*). Blair and Ward employed IR microprofiling to identify surface contaminants as small as 10 μm in diameter. A data reduction program was used to plot three-dimensional projections providing spatial information. A method of coadding spectra from different pixel elements is presented, providing higher quality spectra without increasing data acquisition time (*S2*). Donahue et al. applied chemometric analysis methods to FT-IR microscopy spectra to increase the informational content of spectral data. Factor analysis gives quantitative information and provides pure principal component spectra while reducing the data set and improving the overall signal-to-noise ratio of the compositional maps (*S3*). Windig and Markel describe the use of simple-to-use interactive self-modeling mixture analysis (SIMPLISMA) to FT-IR microscopy data. The method uses a relatively simple algorithm rather than the relatively complicated principal component analysis. All the intermediate steps are displayed in the form of spectra, which makes it possible to use SIMPLISMA interactively in a responsible way without extensive knowledge of the mathematical procedures behind it (*S4*). Pell et al. proposed and tested computational methods that enhance the spatial resolution of IR microspectroscopic data collected from multilayer polymeric materials film structures. Factor analysis of the data matrix can reveal the number of spectrally different layers that are present, and the eigenvectors will give an abstract representation of the positional and wavelength information. An algorithm has been devised that uses layer boundary positions, aperture width, and aperture step size to model the positional information from such an experiment (*S5*).

The technique of IR microspectroscopic functional group imaging has been employed by many investigators to map various materials. Nishioka and Teramae studied coated interfaces and polymer blends (*S6*). Wetzel and LeVine mapped myelin in brains of mice (*S7*). Reffner and Wasacz mapped molecular compounds in biological tissues (*S8*). Kodali et al. employed this technique to probe the subcellular chemical composition of atherosclerotic arterial walls and create three-dimensional images representing changes in composition (*S9*). Louden and Kelly mapped the presence of deterrents (moderants) in nitrocellulose-based propellant grains by FT-IR microscopy (*S10*).

Reffner et al. describe a new IR microscope objective lens for grazing angle FT-IR microscopy. This objective provides a means for the IR analysis of monomolecular films in small (25 × 25 μm) areas. A variety of applications are discussed (*S11, S12*). Eng and Shebib have employed automated FT-IR grazing angle microscopy in studying microspatial chemical mapping of polymer/organic/inorganic films on reflective substrates (*S13*).

Marcott et al. have developed a technique for studying the phase behavior of aqueous surfactant systems using near-IR microspectroscopy. A study of the octyldimethylphosphine oxide/water system illustrates the quantitative potential of this technique (*S14*). Treado et al. have developed an instrumental approach for performing spectroscopic imaging microscopy which integrates and acoustooptic tunable filter (AOTF) and a charge coupled device (CCD) detector with an infinity-corrected microscope for operation in the visible and near-IR spectral regions. Images at moderate spectral resolution (2 nm) and high spatial resolution (1 μm) can be collected rapidly (*S15*).

(T) HYPHENATED TECHNIQUES

Several authors reviewed progress in the area of hyphenated techniques. Fujimoto and Jinno reviewed chromatographic FT-IR spectrometric methods of analysis and discussed the two main approaches to interfacing: flow cell and mobile-phase elimination (*T1*). Gurka reviewed the current status of hyphenated FT-IR techniques (*T2*). Griffiths, Norton, and Lange described a common interface for gas, supercritical fluid, and high-performance liquid chromatographs with eluates deposited on a ZnSe window which is then passed through the microbeam of an FT-IR spectrometer and reported that resulting spectra are similar to KBr disk reference spectra (*T3*). Jinno reviewed microcolumn liquid chromatography FT-IR spectrometry for polymer characterization (*T4*). Griffiths, TeVrucht, and Bet-Pera reviewed TLC/FT-IR and supercritical fluid chromatography (SFC)/FT-IR applied to the analysis of coal extracts (*T5*). Davidson and Jenkins reviewed the capabilities and application of SFC/FT-IR (*T6*). Taylor and Calvey reviewed flow cell SFC/FT-IR (*T7*). Bartle et al. reviewed applications of SFC/FT-IR including the analysis of food, drugs, fuels, and polymers (*T8*). Baumeister and Wilkins reviewed computer enhancement in hyphenated chromatographic/spectrometric techniques including gas chromatography (GC)/FT-IR (*T9*).

(U) MATRIX ISOLATION SPECTROSCOPY

Lui and Davis (*U1*) studied the autoxidation of CF_2CF_2 by condensing the gas-phase reaction mixture into a argon matrix at 10 K. The major product was CF_2. The concentration of CF_2 and other species was found to be dependent upon oxygen concentration and temperature. The absence of CF_2:O as a product illustrated the utility of matrix isolation to quench the reaction and allow identification of transient intermediates. Smith et al. (*U2*) used FT-IR to study the UV laser photolysis products of dicyanoacetylene that had been isolated in argon at 16 K. De Saxce and Schriver (*U3*) studied the photolysis of matrix-isolated chlorine nitrate in argon matrix assisted by reactive solid matrixes of nitrogen and oxygen. Using visible and UV light in the 250–800-nm range, Sodeau and Whyte (*U4*) studied the matrix photo chemistry of CH_3I and CD_3I at 4.4 K. The authors determined that the reaction proceeds mainly through the intermediacy of methylene. Horie and Moortgat (*U5*) followed the reaction products in the oxidation reactions of simple alkenes using matrix isolation FT-IR using a mixture of argon and oxygen as the matrix. The photooxidation of biacetyl was also studied. Dunkin et al. (*U6*) studied the photolysis of 1,2-dinitrosobenzene in argon matrixes at 14 K.

Carpenter and Ault (*U7*) found that the merged jet copyrolysis of mixtures of Ar/B_2H_6 and Ar/MeOH followed by trapping into a cryogenic matrix led to the formation of methoxyborane. Of the 18 fundamentals of methoxyborane, 17 were observed in IR spectra, including the boron–oxygen stretch at 1358 cm^{-1}. Piocos and Ault (*U8*) used matrix isolation to isolate and characterize 1:1 molecular complexes of trimethylgallium with group V(A) bases. Several, including the $(CH3)_3GaSbH_3$ complex, were observed for the first time. Bohn et al. (*U9*) used matrix isolation IR spectroscopy to characterize the cocondensation reaction products of H atoms and CS_2 in Ar at 12 K. Burkholder and Andrews (*U10*) studied the reaction of boron atoms with carbon monoxide. The IR spectrum of BCO, the primary product, was obtained. B(CO)$_2$ (BCO)$_2$ were also identified. Kline et al. (*U11*) studied the reactions of atomic nickel and ethylene oxide in an argon matrix at 12 K using FT-IR spectroscopy.

Hunt and Andrews (*U12*) characterized the HF complexes prepared with CCl_4, $CHCl_3$, and CH_2Cl_2 in argon matrixes using IR spectroscopy. The spectra of the complexes were assigned and discussed. Cesaro and Dobos (*U13*) obtained and discussed the matrix-isolated IR spectra of iron and ruthenium carbonyl cyclopentadienyl complexes and their reaction electrophiles. Radziszewski et al. (*U14*) obtained a matrix-isolated spectrum of tricyclo[3.3.1.03,7]non-3(7)-ene, a doubly pyramidazlized alkene, predicted to have a nearly tetrahedral geometry at the olefinic carbons. Gatia et al. (*U15*) studied the IR and Raman spectra of 1,1,2-trichloro-2,3,3-trifluorocyclobutane, and also the matrix isolation spectra obtained in argon and nitrogen matrixes using the hot nozzle technique. Schallmoser et al. (*U16*) obtained the matrix-isolated spectrum of chlorothioformyl using a pulsed jet dc discharge of Cl_2CS/Ar, CS_2/Cl_2/Ar, and CS_2/CCl_4/Ar. Mosandl and Wentrup (*U17*) obtained the spectrum of bisketene in an argon matrix following the photolysis of benzocyclobutenedione. El-Bindary et al. (*U18*) obtained the conformational equilibria and vibrational spectra, as well as IR matrix isolation spectra of chloroacetyl chloride and chloroacetyl bromide. Smets and Maes (*U19*) studied the vibrational spectrum of 2-hydroxypyridine isolated in an argon matrix using FT-IR. Plitt et al. (*U20*) characterized carbonylchlorocopper isolated in an argon matrix using IR and ab initio calculations.

Les et al. (*U21*) compared the IR spectra of uracil and thymine predicted theoretically at the ab initio Hartree–Fock level with a 6-31G** basis set with argon matrix experimental spectra. Teles et al. (*U22*) studied the flash vacuum pyrolysis and the photolysis of 2,3-thiophenedicarboxylic acid anhydride using matrix isolation FT-IR spectroscopy. No conclusive proof for the existence of 2,3-didehydrothiophene could be found, but two previously unknown isomers were isolated and characterized: vinylidenethioketene and thioformylvinylidenecarbene. Nowak et al. (*U23*) studied the prototropec tautomerism of 2-hydroxypyridine/2(1*H*)-pyridone in low-temperature inert matrixes and in the gas phase. Good agreement between experimental and theoretical predicted frequencies and intensities enabled a reliable assignment of IR bands in the spectra. Nieminen et al. (*U24*) studied the IR spectra of the cis and gauche conformers of 3-fluoropropene in Ne, Ar, Kr, and Xe matrixes. Ab initio calculations were also carried out.

Mielke and Andrews (*U25*) detected the carbonyl phosphine radical and the phosphaketene molecule by reaction of CO with P atoms, or PH radicals produced from P_4 and PH_3 discharge or photolysis. Jiang and Graham (*U26*) identified the fundamental vibration 2, at 2308.6 cm^{-1} for the HCCCO radical, by trapping the vacuum UV photolysis products of a mixture of acetylene and carbon monoxide. The assignment was confirmed by measurements for a variety of D-, ^{18}O-, and ^{13}C-substituted isotopomers. Szczepanski et al. (*U27*) studied the vibrational and electronic spectra of matrix-isolated *p*-dichlorobenzene radical cations and also reported that ab initio self-consistent-field level calculation of the *p*-dichlorobenzene neutral and radical cation species reproduce the spectra well after scaling is performed. Doyle et al. (*U28*) report the identification of a stretching vibration of the hexatriynyl radical at 1953.4 cm^{-1}, after studying the FT-IR spectrum of the vacuum UV photolysis products of acetylene and 1,3-butadiene trapped in solid argon. Szczepanski et al. (*U29*) obtained the IR spectrum of the naphthalene radical cation isolated in an argon matrix. Szczepaniak et al. (*U30*) identified both amino acid–oxo and amino–hydroxy tautomeric forms of 9-methylguanine in IR studies of these molecules isolated in the hydrophobic environment of an argon matrix at 12 K. Thompson and Jacoz (*U31*) reported that IR absorptions attributed to CO^+, trans-$OCCO^+$ and trans-$OCCO^-$ have been recorded when a Ne:CO = 100 mixture is codeposited at 5 K with a beam of neon atoms that have been excited in a microwave discharge.

Xu et al. (*U32*) obtained spectra of aluminum carbonyl complexes generated in argon matrixes by cocondensation of aluminum atoms and carbon monoxide. AlCO was isolated and clearly identified. Szymanska-Buzar et al. (*U33*) obtained and studied spectra of intermediates in the photochemical reaction of tungsten hexacarbonyl with carbon tetrachloride. Feltrin et al. (*U34*) studied gaseous cerium dicarbide species isolated in argon. For the first time, experimental evidence was obtained for the presence of a C═C bond in the structure of a gaseous rare-earth dicarbide species. Biggs et al. (*U35*) characterized molecular alkali metal iodates by mass spectrometry and matrix isolation IR spectroscopy in N, O, and Ar matrixes. The molecular species KIO_3, $RbIO_3$, and $CsIO_3$ were identified. Mossoba et al. (*U36*) identified minor C_{18} and conjugated diene isomers in hydrogenated soybean oil and margarine by GC/MI-FT-IR spectroscopy. Wilson et al. (*U37*) used matrix isolation infrared spectroscopy to determine polycyclic aromatic hydrocarbons in diesel exhaust particles and urban air particulate samples.

(V) GC/FT-IR

Saggese et al. reported the design- and temperature-dependent mid-infrared transmission characteristics for gold and silver on nickel, silica, and sapphire hollow lightpipes and discussed their advantages compared to conventional FT-IR lightpipes (*V1*). Diederich et al. compared conventional lightpipe GC/FT-IR with the tracer GC/FT-IR for the analysis of perfumes and reported improved sensitivity for the cryotrapping technique of the tracer with spectra similar to condensed-phase spectra (*V2*). Cole-Clarke and Powell applied a cryotrapping interface for GC/FT-IR to the analysis of environmental samples (*V3*). Powell and Compton used cryotrapping GC/FT-IR to characterize flavors and fragrances in beers derived from different brewing processes (*V4*). chromatography Visser et al. (*V5*) and Vredenbregt et al. (*V6*) applied cryotrapping GC/FT-IR to the analysis of compounds in bovine urine and liver extracts and photolysis products of chloronitrobenzenes. Smyrl et al. used a cold trapping interface in a combined GC/FT-IR/GC/MS instrument for the analysis of complex mixtures (*V7*). Williams et al. evaluated a combined GC/FT-IR/MS system for the identification and quantitation of 50 phenolic compounds (*V8*). Haaland et al. reported the use of classical least squares techniques (CLS) as a method superior to coaddition of spectra in a single-component peak for improved S/N ratio of the spectrum of the eluted analyte obtained by GC/FT-IR. They also reported that increasing the number of background scans to sample scans is a convenient method for improving S/N of the composite spectrum obtained by either method (*V9*). Pyo and Harada demonstrated computer methods for selecting ranges of frequencies for characteristic group frequencies allowing the computer to serve as a selective detector for functional groups (*V10*). Jarvis and Kalivas described a procedure for library searching and identification of components detected by GC/FT-IR without complete chromatographic separation (*V11*).

Filippelli et al. reported a method for determining methylmercury as methylmercury hydride by purge-and-trap GC/FT-IR spectrometry (*V12*). Compton and Stout used headspace GC/FT-IR to analyze flavor components in cinnamon, banana cream, and apricot oils (*V13*). Compton et al. applied GC/FT-IR and headspace GC/FT-IR to the analysis of food and food packaging materials including beer in cans and bottles (*V14*). Jirovetz et al. used GC/FT-IR to identify volatile components in German onion oil and Roselle seed oil (*V15*, *V16*). Hedges and Wilkins characterized eucalyptus oil using GC/FT-IR/MS (*V17*). Kalasinsky et al. discussed the utility of cryogenic sample deposition GC/FT-IR interfaces for the analysis of drugs (*V18*). Platoff et al. reported the application of GC/FT-IR/MS to the qualitative and quantitative analysis of amphetamine, methamphetamine, and related compounds in human urine (*V19*). Soine et al. evaluated GC/FT-IR/MS for the identification of amphetamine isomers and homologs (*V20*). Kurz et al. applied GC/FT-IR to the analysis of alkyl nitrites (*V21*). GC/FT-IR was used to characterize plasticizer levels in solid propellants by Pesce-Rodriguez and Fifer (*V22*). Ethers and alcohols were analyzed in gasoline samples by Diehl et al. using GC/FT-IR (*V23*). Zhang et al. used GC/FT-IR to characterize light oil fractions of coal tar (*V24*). Semmler et al. applied GC/FT-IR to the analysis of 33 polynuclear aromatic hydrocarbons (*V25*). The characterization of isomers of polychlorinated biphenyls using GC/FT-IR was reported by Doumenq et al. (*V26*) and by Hembree et al. (*V27*) using GC/FT-IR/MS. GC/FT-IR was used to characterize isomers of *p*-nonylphenols by Bhatt et al. (*V28*). Sojak et al. applied GC/FT-IR to the identification of isomers of *n*-nonadecenes (*V29*). Gurka et al. reported the analysis of water-soluble environmental contaminants by direct aqueous injection GC/FT-IR and GC/ion trap spectrometry (ITS) (*V30*). Philippaerts et al. discussed the application of purge-and-trap GC/FT-IR for the analysis of wastewater samples (*V31*). Veijanen et al. used GC/FT-IR to determine off-flavor compounds produced by algae in freshwater systems (*V32*). Dworzanski et al. reported the interfacing of microreactors to GC/IR/MS for pyrolytic degradation studies of jet fuels in the vapor phase and under supercritical conditions (*V33*). Zunic et al. applied pyrolysis GC/FT-IR to the characterization of peat water samples (*V34*, *V35*). Weber et al. analyzed industrial poly(ester urethane) elastomers by pyrolysis GC/FT-IR (*V36*). Pesce-Rodriguez et al. studied pyrolysis products of low-vulnerability ammunition propellant formulations using GC/FT-IR (V37). The application of GC/matrix isolation (MI)/FT-IR to the analysis of environmental air samples was evaluated by Childers et al. (*V38*). GC/MI/FT-IR applied to the characterization of essential oils was reported by Coleman and Gordon (*V39*).

Marchand et al. (*V40*) studied mixtures of cyclosilazanes, -siloxanes, and -siloxazanes (six- and eight-membered rings) using low- and high-resolution electron impact mass spectroscopy/gas chromatography and FT-IR gas chromatography. Pesce-Rodriguez et al. (*V41*) reported on pyrolysis GC/FT-IR studies of a LOVA propellant formulation series, with the primary objective of identifying correlations between ignition times and pyrolysis product distributions. The most noteworthy correlation observed was between the amount of permanent gas produced and go/no-go ignition times. The results of the study may serve as a basis for formulation design and bench-scale propellant screening. Kuric et al. (*V42*) identified various volatile radiolytic products formed in the γ-radiolysis of *o*-, *m*-, and *p*-dinitrobenzene solution in carbon tetrachloride using a combined system of GC/FT-IR/MS. Kuric et al. (*V43*) also reported using the technique to determine the volatile radiolysis products of nitrophenol solutions in carbon tetrachloride. Hasenoehrl et al. (*V44*) described a procedure for the on-line characterization of compounds eluting from a gas chromatograph using automated interpretation of their vapor-phase IR spectra. Principal component analysis was applied to a training set containing two classes of appropriately scaled vapor-phase IR library spectra. Guisto et al. (*V45*) reported temperature-programmed retention indexes for 17 industrially important adipate esters along with mass and IR spectra for several of the adipate esters. Kubwabo et al. (*V46*) identified eight alkaloids in the aerial parts and roots of *Physalis peruviana* using capillary GC, capillary GC/MS, and GC/FT-IR.

(W) LC/FT-IR

Kalasinsky and Kalasinsky reviewed the design and application of HPLC/FT-IR (*W1*). Somsen et al. developed a solvent elimination interface for reversed-phase HPLC/FT-IR using a spray jet assembly and reported detection limits in the 10–20-ng range and application with eluents containing up to 40% water (*W2*). Somsen et al. demonstrated the utility of the spray jet interface for analyzing impurities in steroids (*W3*). Griffiths and Lange developed an interface for microbore reversed-phase HPLC/FT-IR spectrometry, using a concentric flow nebulizer to eliminate the mobile phase with analytes deposited on a moving ZnSe window, and reported that eluents containing up to 100% water could be used with this interface (*W4*). Lange and Griffiths demonstrated that the interface could be used with mobile phases consisting of 63–100% water containing volatile and nonvolatile buffers. Spectral subtraction was used to obtain analyte spectra in the nonvolatile buffered systems (*W5*). Raynor, Bartle, and Cook reported the development of a microcolumn HPLC/FT-IR microspectrometry interface using electrospray nebulization. The column effluent is subjected to a relatively high positive potential and the electrical field generated causes the liquid to disintegrate into charged droplets. Nitrogen gas is used to evaporate solvent from the droplets as they are attracted to a ZnSe plate where the analytes are deposited. Total elimination of the solvent is possible with low flow rates, and spectra obtained from less than 500 pg of sample are reported for caffeine as a test compound (*W6*). Robertson et al. continued the development and optimization of a thermospray interface applicable to both normal and reversed-phase HPLC/FT-IR, which thermally desolvates the HPLC effluent and deposits analytes on a moving metal substrate used with a DRIFT accessory of an FT-IR spectrometer. They applied the interface to the analysis of various compounds and reported the identification of Irganox 565 antioxidant at concentrations below 50 μg/mL (*W7*).

DiNunzio reported the application of a solid-phase extraction interface HPLC/FT-IR system to the identification of degradation products and isomers in active ingredients, and contaminants in pharmaceuticals, and described the advantages of the extractor for preconcentration to increase sensitivity (*W8*). Meyer et al. applied HPLC/FT-IR using a flow cell interface to identify reactive diluents in epoxy resins and reported that limits of detection for the on-line system are at least 1 order of magnitude higher than those obtained off-line using DRIFTS after evaporation of the solvent (*W9*). Sreenivasan developed an HPLC/FT-IR method to analyze the antioxidant in biomedical polyurethane (*W10*). Robertson et al. applied HPLC/FT-IR to the analysis of propylenediol adipate polyester and determined that early eluting fractions contained only one isodecyl end cap while later eluting fractions were doubly end capped (*W11*). Markovich et al. applied gel permeation chromatography (GPC)/FT-IR spectrometry to the characterization of ethylene-based polymers (*W12*).

Patonay and Czuppon reviewed HPLC detection in the near-infrared and discussed the advantages of using near-IR-absorbing chromophores as labels in HPLC separations, including lower detection limits and lower background interference. They reported that HPLC/near-IR is becoming affordable with significant development of semiconductor laser diodes as light sources, but that there is a lack of commercially available near-IR-absorbing labels (*W13*). Williams et al. compared noncovalent and covalent methods of labeling protein with near-IR dyes to analyze human serum albumin by HPLC/near-IR (*W14*).

De la Guardia et al. (*W15*) and Guzman et al. (*W16*) investigated the interfacing of flow injection analysis (FIA) with FT-IR spectrometry using transmittance flow cells and a cylindrical internal total reflectance (CIRCLE) cell. Tilotta et al. used cross-flow pneumatic and thermospray nebulizers to interface HPLC with flame infrared emission (FIRE) spectrometry (*W17*). Lam et al. reported the design of a low-volume purge cell to interface ion exchange HPLC/FIRE and FIA/FIRE spectrometry (*W18*).

(X) TLC/FT-IR

Frey, Kovar, and Hoffmann evaluated alternatives for quantitative analysis of components separated and detected by TLC/FT-IR and reported that using peak areas in Gram–Schmidt chromatograms was the best universal method for quantitation based on precision, selectivity, and time involved (*X1*). Wada et al. (*X2*) and Tajima et al. (*X3*) reported a zone-transfer technique for TLC/FT-IR with components separated by TLC transferred to a KBr powder layer on the TLC plate, which is then placed in a diffuse reflectance attachment with a scanning sample stage. Kovar et al. discussed the application of TLC/FT-IR to the identification and quantitation of complex mixtures, including designer drugs (*X4*). Danielson et al. reported the use of zirconium oxide as a stationary phase for TLC/FT-IR using in situ diffuse reflectance IR microspectroscopy (*X5*). Yamamoto et al. analyzed phospholipids by Fourier transform near-IR spectrometry on TLC plates placed on a microscope stage linked to the spectrometer (*X6*). Petty and Cahoon investigated the use of near-IR FT-Raman to record spectra of components separated by TLC directly from the plates with no special treatment of the samples or plates required (*X7*).

(Y) SFC/FT-IR

Jenkins et al. give an overview of hyphenated techniques involving supercritical fluids including SFC/FT-IR applied to quantitative characterization of polymeric surfactants, and supercritical fluid extraction (SFE) followed by SFC/FT-IR (*Y1*). Griffiths et al. described the flow cell and solvent elimination methods of interfacing chromatographs and FT-IR spectrometers and discussed the interfacing of SFC with FT-IR spectrometry by elimination of the mobile phase (*Y2*). Jenkins et al. described a flow cell interface for capillary supercritical fluid chromatography (cSFC)/FT-IR and reported improved detection limits over literature values for a range of compounds including pyrene (2 ng) and caffeine (95 pg) (*Y3*). Sheu et al. developed an automated interface between an SFC microextractor and a micro-FT-IR spectrometer to characterize low molecular weight fractions of asphalt (*Y4*). Fuoco and Griffiths applied SFE-SFC/FT-IR to the analysis of PCBs in contaminated soil (*Y5*). Kirschner and Taylor developed an SFE/FT-IR system for the quantitative analysis of *n*-tetracosane (C24) in Celite and have demonstrated its utility with other systems. The interface uses a flow cell and stainless steel transfer line directly from the SFE extraction vessel/system cryotrap without prior chromatographic separation (*Y6*).

Jenkins et al. compared xenon and carbon dioxide as mobile phases for SFC/FT-IR using compounds in a range of polarities and compared SFC/FT-IR spectra with vapor- and condensed-phase spectra. They investigated the effects of mobile-phase temperature and density on infrared spectra and reported that carbon dioxide and xenon had similar effects and that carbon dioxide had smaller shifts with changes in temperature and density than expected (*Y7*).

(Z) TGA/FT-IR

Mullens et al. discussed the capabilities of interfacing thermogravimetric analysis (TGA) and FT-IR spectrometry (*Z1*). Compton et al. combined TGA with differential scanning calorimetry (DSC) in a simultaneous on-line FT-IR analysis of the evolved gases to minimize operator time and resolve differences that can result from separate analyses (*Z2*). Jansen et al. developed a TGA/FT-IR interface with the IR beam led directly into the TGA and reflected back by a mirror mounted inside the analyzer (*Z3*). Chen and Smart reported the application of TGA/FT-IR to analyze components separated by thin-layer chromatography which were scraped from silica gel TLC plates and loaded into the TGA sample pan (*Z4*).

Applications of DSC/FT-IR to investigate cross-linking and thermal transitions in polymer systems were reported by Johnson et al. (*Z5*). Clark and Gray applied TGA/FT-IR to obtain quantitative real-time data for decomposition studies of a polymeric foam (*Z6*). Mullens et al. used TGA/FT-IR to compare oxidative degradation of polystyrene samples containing different additives (*Z7*). Kinoshita and Teramoto applied combined TGA/DTA/FT-IR spectrometry to investigate thermal degradation of poly(ethylene terephthalate) and poly(butylene terephthalate) (*Z8*). Bowley et al. applied TGA/FT-IR to determine the percentage of butadiene and styrene in unknown composite samples (*Z9*). TGA/FT-IR was applied to the analysis of ethylene–vinyl acetate copolymers by Maurin et al. (*Z10*). Roth et al. used TGA/FT-IR to investigate combustion profiles of various blends of refuse-derived fuels, coal, and limestone (*Z11*). Cai and Smart applied TGA/FT-IR to the quantitative analysis of *N*-methyl-2-pyrrolidinone retained in coal extracts (*Z12*). TGA/FT-IR was used by Shao et al. (*Z13*) to study chlorine released from coal during pyrolysis. Solomon et al. applied TGA/FT-IR to the analysis of coal samples, obtaining kinetic rates for pyrolysis modeling (*Z14*).

(AA) SPECTRAL LIBRARIES, SEARCHING, COMPUTER-ASSISTED INTERPRETATION, ARTIFICIAL INTELLIGENCE, AND DATA TRANSFER

De Koeijer et al. have developed an automated system for the qualitative analysis of polymers using IR data (*AA1*). De Koeijer et al. have also developed a knowledge-based system for the characterization of copolymers based on IR data (*AA2*). Luinge et al. have applied univariate and multivariate calibration methods for the quantitative analysis of aryl ether sulfone–aryl ether sulfone copolymers (*AA3*).

Tungol et al. have developed an electronic data base for the identification of fibers whose spectra have been recorded using IR microscopy techniques (*AA4*). Heller has reviewed the computerized spectroscopy data bases for IR, Raman, NMR, and ESR (*AA5*). Pasquim and Davanzo have developed a program for expanding IR data files from a Perkin-Elmer FT-IR1600 (*AA6*).

Bruno has published a spectroscopic library for IR and MS spectra for chlorofluorobromoethanes and ethylenes (*AA7*). Prestrelski et al. have developed a substructure library for the description and classification of protein secondary structure (*AA8*). Jarvis and Kalivas have developed a procedure for library searching GC/FT-IR spectra (*AA9*).

Lo and Brown have developed a new algorithm for searching medium-size and large-size IR spectral libraries for the components in spectra of mixtures (*AA10, AA11*). Tomellini et al. reviewed knowledge-based systems for interpreting IR spectra (*AA12*). Liu et al. have developed an artificial neural network system for estimating composition from chemometric near-IR and Raman data (*AA13*). Smits et al. developed modular neural network systems for the interpretation of IR spectral data (*AA14*).

Bruchmann et al. have developed an IR spectral search system based on Hamming networks. This system is reported to identify spectra of mixtures with high fault tolerance (*AA15*). Meyer and Weigelt have developed artificial neural networks for the interpretation of IR spectral data based on a Pascal program which was implemented on a PC (*AA16*). Fessenden and Gyorgyi have developed a program for identifying functional groups present in molecules via neural networks and IR spectral data (*AA17*).

Tanabe et al. have developed a neural network system on a PC to identify molecule structure via IR data (*AA18, AA19*). Debska developed the Scannet method for the identification of molecular structure based upon six different spectral methods: IR, MS, Raman, UV, ^{13}C NMR, and ^{1}H NMR (*AA20*).

Rothman et al. discussed the modification, improvement, and enhancement to the HITRAN molecular data base for the identification of molecular structure (*AA21*). Pyo and Griffiths have developed a computerized expert system for the interpretation of vapor phase IR spectra (*AA22*). Cabrol et al. have developed a computer-assisted program to help teach students how to interpret IR data (*AA23*). Pyo demonstrates a moving pattern search system in the selective reduction of IR data (*AA24*). Hasenoehrl et al. have developed an expert system for the rapid functional group characterization of GC/FT-IR spectra by principal component analysis (*AA25*).

Pyo et al. have developed an expert system for the identification of vapor-phase IR spectra of aromatic compounds (*AA26*). Weigel and Herges have developed a system for the automatic recognition of the aromatic substitution pattern (*AA27*). The project committee has recommended the use of JCAMP-DX to standardize IR spectral data for ease in transmission between IR spectrometer data systems and between such systems and other computers (*AA28*).

Debska has developed a multimethod (IR, NMR, MS, RA, UV) data base and search system for identifying chemical compositions (*AA29*). Jin et al. have developed an expert system to interpret IR spectra using an expert system shell (*AA30*).

Kvall et al. have studied the performance of artificial networks in near-IR spectroscopy (*AA31*). Lebedev et al. have discussed a computerized method of recognizing large structural fragments of an unknown by its IR spectrum (*AA32*). Kavak and Esen have developed a group of algorithms and a computer program based on these algorithms for comparing IR spectra of different origins (*AA33*). Volmer et al. used partial least squares regression of FT-IR data in the determination of urinary calculus composition (*AA34*).

(BB) THEORETICAL STUDIES, BAND INTENSITIES, VIBRATIONAL ASSIGNMENTS, TEMPERATURE EFFECTS, AND PRESSURE EFFECTS

Gulaubov et al. measured the IR band intensities for toluene, *p*-xylene, and benzene. The signs of the intensity parameters were determined by 4-31G ab initio molecular orbital calculations (*BB1*). Fan and Ziegler applied density functional theory to IR absorption calculations on H_2O, NH_3, H_2CO, C_2H_4, CH_3OH, and oxirane (*BB2*). Bertie et al. have used a soundly based procedure to measure the absolute intensity of the 1036-cm^{-1} band of benzene (*BB3, BB4*). Figeys and Geerlings have written a review on the aspects of the quantum chemical interpretation of integrated intensities of IR absorption bands (*BB5*). Emmence and Steele have explored the use of factor analysis for determining the band intensity distribution arising from specific functional groups in a molecule (*BB6*). Bertie et al. have reviewed the progress in obtaining standards for calibrating IR band intensity measurements of materials in the liquid phase (*BB7*). Le Sueur et al. have used variational wave functions in calculating IR band intensities of molecules (*BB8*). Lii and Allinger have added a quality intensity calculation to the MM3 molecular mechanics program which is used to obtain a fair representation of vibrational frequencies for molecules (*BB9*). Berezin and El'kin have discussed a valence-optical scheme (Volkenstein–Elyashevitch–Stepanov scheme) and the Mayants–Averbukh method as semiempirical models for the theory of band intensities of rotation/vibration spectra of polyatomic molecules (*BB10*).

Bonatsos and Daskaloyannis have used deformed bosons to construct generalized anharmonic oscillators suitable for the description of vibrational spectra of diatomic molecules (*BB11*). Tennyson has reviewed the literature pertaining to the uses made of rovibrational calculations on small molecules, particularly triatomic molecules (*BB12*).

Feldman et al. obtained a least squares fit of a short-range-force constant mode to the 14 Raman and IR active normal-mode frequencies of C60 fullerene (*BB13*). Huang reviewed the IR and Raman conformational studies of a series of 10 dinitroethane derivatives (*BB14*). Makarewicz has discussed the effective Hamiltonians used in the calculations and analysis of vibrational spectra (*BB15*). Ramondo et al. have reported on the ab initio calculations at the split-valence basis set level on inorganic anions and radical anions and on the coordination structures with alkali metal cations (*BB16*). Procacci et al. have proposed a vibration potential function for C70 fullerene in terms of stretching, bending, and nonbonded interactions (*BB17*).

Duval has derived the selection rules for far-IR and Raman transitions of a solid sphere using group theory (*BB18*). Van Duijneveldt has performed ab initio calculations on the structures and hydroxyl vibrations on small hydrogen-bonded systems such as $(H_2O)_2$, $(CH_3OH)_2$, H_2O-H_2CO, and $(H_2O)_3$ (*BB19*). Ha et al. have performed ab inito calculations on the vibrational frequencies of the trans-trans and trans-gauche conformers of ethyl acetate (*BB20*). Fernandez et al. have made theoretical studies on 3-chloro-, 3-hydroxy-, 3-mercapto-, and 3-aminopropenitriles and 4-chlorobutanenitrile (*BB21*).

Coolidge has reported on the calculations of molecular vibrational frequencies using semiempirical methods (MINDO/3, NNDG, AMI, and PM3 calculations) for 61 molecules (*BB22*). Cheam has performed ab initio calculations for hydrogen-bonded *N*-methylacetamide (*BB23*), and Mirkin and Krimm have performed ab initio calculations for hydrogen-bonded *cis*- and *trans*-*N*-methylacetamide (*BB24*). Mohan and Sundaraganesan have made assignments and a normal coordinate analysis of *N*-methylacetamide (*BB25*). Oestergaard et al. have made an ab initio investigation of unassociated and hydrogen-bonded formamide (*BB26*).

Crowder and Carlisle have assigned the vibrational data for 1,2-dibromopropane and its D-6 analog (*BB27*). Kwiatkowski and Leszczynski have performed ab initio SCF-31G calculations on phosphine oxide, phosphine sulfide, and their fluoro derivatives (*BB28*). Taga has assigned the vibrational frequencies for the rotational isomers of dimethyl phosphorochloridate and trimethyl phosphate (*BB29*). Durig et al. have performed ab initio calculations for dimethyl (methylthio)phosphine (*BB30*).

Faria et al. have obtained variable-temperature spectra of methyl *trans*-crotonate and assigned the spectra for the s-cis and s-trans conformers (*BB31*). Nieminen et al. have matrix-isolated 3-fluoropropene and performed ab initio studies on its conformers (*BB32*). Ma et al. have assigned the vibrational data for tetraethynylethylene (*BB33*). Durig et al. have performed ab initio calculations for the alkyl halides and ethers (*BB34*).

Rogojerov and Arnaudov have developed a method of Fourier deconvolution of IR spectra using a reference band (*BB35*). Manolopoulos has developed a general and convenient method by which the point group, the ^{13}C NMR pattern, and the number of IR and Raman active vibrations of any given fullerene structure can be obtained from its molecular graph (*BB36*).

Snyder has developed a method to quantitate information about conformation in assemblies of feasible chains in liquid *n*-alkanes C12–C20 (*BB37*). Durig et al. have performed ab initio calculations for 3-iodopropene (*BB38*). Karlsson et al. have assigned the vibrational spectra of 1,4-dibromo-but-2-yne and 1,4-diiodobut-2-yne (*BB39*). Durig et al. have performed ab initio calculations for *trans*-1,3-dichloroprene (*BB40*).

El-Bindary et al. have studied the conformational equilibria and matrix isolation spectra of bromoacetyl chloride and bromoacetyl bromide (*BB41*) and for chloroacetyl chloride and chloroacetyl bromide (*BB42*). Durig et al. have written a review on the conformational stability of the haloacetyl halides (*BB43*). Durig et al. have performed ab initio calculations for 2-methylpropionyl fluoride (*BB44*). Francisco and Williams have made an experimental and theoretical study of the IR and UV spectra of trifluoroacetyl fluoride and trifluoroacetyl chloride (*BB45*).

McKean has surveyed the assignments of the SiH, SiD modes in $SiHX_3$, SiH_2X_2, and SiH_3X compounds (*BB46*). Bramley have used a refined quadratic force field for acetylene in order to accurately calculate its vibrational spectrum (*BB47*).

Stuchebryukov and Rudoi have considered the theoretical aspects of the contact problem in ATR/FT-IR (*BB48*). Tickanen et al. have determined the optical contents and the Kramers–Kronig integration constant (the anchor point) using variable angle ATR/FT-IR (*BB49*). Gu has used factor analysis as an aid in the analysis of IR spectra recorded of pentaerythritol tetrastearate at various temperatures (*BB50*).

Nyquist et al. have compared the vibrational assignments of α and β syndiotactic, isotactic, and atactic polystyrene and toluene (*BB51*). Dybal performed a normal coordinate analysis on the vibrational data for syndiotactic poly(methyl methacrylate) (*BB52*). Kofranek, Lischka, and Karfen have performed ab initio calculations on the structure and vibrational spectra of all-trans polyenes ranging from C_4H_6 to $C_{18}H_2O$ (*BB53*).

Oss et al. developed a new code for computing vibrational levels of general triatomic molecules in the framework of an algebraic model (*BB54*). Bakker and Lagendijk performed a time-resolved study of the vibrational relaxation of the C–H vibration for dilute solutions of $CHBr_3$ and $C_2H_2Br_4$ and the pure liquid $CHBr_3$ (*BB55*). Barron et al. obtained experimental data and performed ab initio theoretical calculations on the optical activity of tartaric acid (*BB56*).

Guirgis et al. recorded the IR and Raman spectra of the difluoroacetyl halides and performed ab initio calculations using the recorded data and vibrational assignments (*BB57*). Davis et al. used the combination of Raman data and ab initio calculations to determine the conformational stability of the oxalyl halides (*BB58*). Durig et al. used Raman and far-IR data to confirm the structure of 2,3-dichloropropene and both *cis*- and *trans*-1,3-dichloropropene (*BB59*). VanDerVeken et al. performed a vibrational analysis of both methyl fluoroacetate and methyl difluoroacetate (*BB60*).

Wong used pressure-tuning IR spectroscopy for the study of structural and dynamic properties of membranes at the molecular level. These data are reported to be useful in cancer research and diagnosis (*BB61*).

Durig and Wang discussed ab initio calculations in vibrational spectroscopy for molecules with 12 or more atoms using the 3-216 basis set (*BB62*). Krutules et al. used vibrational data (IR and Raman) and ab initio calculations to determine the conformational stability for fluorocarbonyl and acetyl isocyanate (*BB63*). Durig et al. recorded vibrational data, made assignments, and performed ab initio calculations for cyclopropylmethylsilanes (*BB64*).

Chong and Bree calculated the IR band intensities for the bending mode in some small molecules (*BB65*). Majoube and Vergoten made vibrational assignments for uric acid based on normal coordinate analysis (*BB66*). Rosas et al. made a conformational analysis for isopropyl formate using vibrational data (*BB67*). Hess made an ab initio study of the structure and vibrational spectrum of allene oxide (*BB68*).

Gejji et al. performed an ab initio calculation on the vibrational frequencies of triflic acid (*BB69*). Ishida and Girffiths compared the amide I/II intensity ratio of the solution and solid state of albumin (*BB70*). Durig et al. used vibrational data to determine the conformational stability and barriers to internal rotation for acetyl isocyanate (*BB71*) and 2-bromo-3-fluoropropene (*BB72*).

Gatial et al. made vibrational assignments for 2,2-bis-(fluoromethyl)-1,3-difluoropropane based upon ab inito force fields (*BB73*). Karlsson et al. assigned the vibrational spectrum of 2,3-diiodobuta-1,3-diene (*BB74*).

(CC) GROUP FREQUENCIES, SOLUTE/SOLUTION STUDIES, SPECTRA/STRUCTURE CORRELATIONS, AND QUALITATIVE PRACTICES

Nyquist et al. have studied the solvent effects of several compounds in $CHCl_3$ and/or CCl_4 solutions by application of IR. These are as follows: alkyl isocyanates (*CC1*), 4- and 3-substituted anilines (*CC2*), trialkyl-1,3,5-triazine-2,4,6-(1*H*,3*H*,5*H*)trione (*CC3*), phosphorus oxychloride and organophosphorus compounds containing the $P(=O)Cl_2$ group (*CC4*), benzaldehyde and 4-substituted benzaldehydes (*CC5–7*), tetraalkylureas (*CC8*, *CC9*), dimethylacetamide (*CC10*), alkyl acetates (*CC11*), 14*H*-dibenzo-1,*j*-xanthen-14-one (*CC12*), 1,4-diphenylbutadiyne (*CC13*), and 1,4-benzoquinone and substituted 1,4*x*-benzoquinones (*CC14*). References CC3, CC13, and CC14 also contain Raman data. References CC3, CC7–9, CC11, CC12, and CC14 also contain data for other solvents beside $CHCl_3$ and CCl_4. Nyquist has developed IR and NMR correlations for alkyl isocyanates (*CC15*) and anisoles (*CC16*).

Benavente et al. have studied solvent affects on IR and ^1H NMR spectra of N,N′-thiodianilines (*CC17*). Hoba and Condrea have developed correlations between IR and NMR data in binary mixtures (*CC18*). Kolling has reported on the carbonyl stretching frequency of ethyl acetate in aprotic solvents (*CC19*).

Eaton et al. have studied the carbonyl stretching frequency of amides in mixed-solvent systems and compared the IR data with calorimetric measurements (*CC20*). Wong et al. studied the tautomeric equilibria of formamide and 2-pyridone in the gas phase and in solution (*CC21*). Faguy et al. have studied ion/solvent and ion/ion interaction in lithium perchlorate/nitromethane solutions by application of ATR/FT-IR (*CC22*).

Castilho et al. studied substituent and solvent effects for substituted nitrophenols by application of IR (*CC23*). Denisov et al. studied the NH_2 stretching frequencies of ortho-substituted anilines with proton acceptors (*CC24*). Schreiber and Shchepkin used IR to study the solvent effect on a hydrogen-bonded complex (*CC25*).

Epstein et al. utilized IR to study hydrogen bonding with anions in low-polarity and nonpolar solvents (*CC26*). Linder studied the effect of solvent on integrated band intensities (*CC27*). Tachikawa et al. performed a theoretical study on solvation effects in chemical reactions using vibrational coupling as a model (*CC28*). Marcott et al. discussed how the information from IR and Raman data is enchanced by sample perturbation (*CC29*).

Juchnovski et al. have reported on the C–O stretching frequencies of benzophenone ketyls (*CC30*). Beckenkamp and Lutz have reported on the OH stretching frequencies of OH ions in the spectra of solid hydroxides (*CC31*). Yukhnovski et al. have stated that there are systematic deviations of the CN stretching frequency of *m*- and *p*-benzonitriles whose spectra have been recorded in a variety of solvents (*CC32*).

Berthelot et al. have correlated the OH stretching frequency of isoborneols with the chemical atoms or groups substituted in the 4-position (*CC33*). Venkateshwarlu et al. have reported that ring-substituted phenyl chloroacetates exist as cis and gauche rotational isomers in solution (*CC34*). Kolb et al. have reported on the relationship between the carbonyl stretching frequencies of semicarbazones and their solid-state structures (*CC35*).

Dessouki et al. have reported IR and ^1H NMR data in different solvents for azo–azomethine dyes (*CC36*). Passingham et al. have reported on the NO_2 stretching frequencies of nitrobenzenes using Raman spectroscopy (*CC37*).

Jamroz and Dobrowolski have determined that the dihalomethanes form double hydrogen bonding with electron donor organic solvents by application of IR spectroscopy (*CC38*). Pappalardo et al. have studied solvent effects on molecular geometries and the isomerization processes (*CC39*).

Nyquist has developed IR and NMR correlations for 3-*x* and 4-*x* substituted anilines (*CC40*). Liu concluded that the vibrational frequencies of crystals depend solely on variation of the volume with change in pressure and temperature (*CC41*). Kolb et al. have developed a relationship between IR C=O stretching frequencies and their solid-state structures (*CC42*). Nyquist studied solute/solvent interactions between separate chloroform solutions of alkanes, cycloalkanes, alkenes, cycloalkenes, cyclohexadienes, benzene, and carbon tetrachloride (*CC43*). Nyquist and Puehl studied alkyl isothiocyanates in carbon tetrachloride and/or chloroform solutions (*CC44*).

Ikram et al. studied the effects of solute/solvent interactions on IR spectra of dimethyldichlorosilane in a number of solvents (*CC45*). Bekarek and Hritzova studied the solvent effect on the N–H stretching frequency of N-substituted *N*-1-(2-chlorobenzoyl)thioureas (*CC46*). Brunner et al. correlated the proton NMR chemical shift and the OH stretching frequency of hydroxyl groups in solids (*CC47*).

(DD) FIBER OPTICS

Fiber-optic spectroscopy has been widely practiced in the near-IR region for many years, and in fact, most of the applications of near-IR spectroscopy result from the ease with which these measurements can be made remotely through silica core optical fibers. The practice of mid-IR measurements has been hampered by the availability of rugged, transparent optical materials; however, short lengths of various materials are being used for some, mostly laboratory, applications.

Saggese and co-workers investigated the use of hollow waveguides for sensor applications (*DD1*). Ordered bundles of silver halide crystalline fibers were fabricated by Paiss and Katzir by extrusion of crystalline preforms for the purpose of thermal imaging (*DD2*). Chalcogenide glass optical fibers were used to direct IR radiation into a zinc selenide circle cell by Moser et al. in order to study the hydroformylation of olefins (*DD3*). Zinc selenide coated silver waveguides were investigated for the purpose of transmitting light from a carbon dioxide laser by Matsuura and Miyagi (*DD4*). Driver and co-workers described applications of multiplexed sensor systems in quantitative IR spectroscopy (*DD5*). Burger and co-workers described the use of mid-IR fiber-coupled laboratory apparatus for remote spectroscopy (*DD6*). Zirconium fluoride optical fibers were used in a fiber-optic accessory for FT-IR studies reported by Smith and May (*DD7*). The surface modification of a chalcogenide glass fiber by chemically bound enzymes was used for the construction of a fiber-optic chemical sensor by Taga and Kellner (*DD8*). Bernard and co-workers received a patent for a fiber-optic-based attenuated total relection probe for the mid-IR (*DD9*).

One means of circumventing the mechanical instability problems of mid-IR-transmitting optical fibers is accomplished by stripping the cladding off the fiber for a short distance and immediately embedding it in an analyte, most often a polymer undergoing curing. This technique utilizes the evanescent field effect with the fiber itself as the internal reflection element. Examples of the theory and application of such spectroscopy are given in references (*DD10–14*).

(EE) MISCELLANEOUS INFRARED TECHNIQUES

McClelland and Jones describe a method and apparatus for transient thermal IR spectrometry of flowable enclosed material. Transient IR emission and transmission spectroscopies are a pair of closely related techniques which give useful spectra from opaque, moving materials. In the techniques, a transient thermal gradient is used to spectroscopically isolate a thin surface layer from the bulk of the sample. The basic elements of the techniques and some industrially relevant applications are described (*EE1*, *EE2*). Osawa et al. demonstrate that the IR absorption of molecules is greatly enhanced (101–103 times) when they are absorbed on or near vacuum-evaporated Ag or Au thin films. By using this surface-enhanced IR absorption (SEIRA) phenomenon, the authors could obtain IR spectra of some organic molecules of a few-picogram amount in good signal-to-noise ratios. Thus, the SEIRA spectroscopy is promising as a new trace analysis technique. General properties of SEIRA found in the experiments are summarized. An electromagnetic theory of this phenomenon is proposed (*EE3*). Nishikawa et al. identified subnanometer surface layers on polymer films, which were only analyzable by XPS or SIMS, by using transmission SEIRA spectroscopy and Ag island films. Dimethylsiloxane (PDMS, 1.0 nm thickness) film or methylphenylsiloxane (PMPhS, 0.5 nm thickness) surface layers on PET films were successfully identifiable with SEIRA by measuring the PDMS or PMPhS that was transferred onto the Ag-deposited BaF_3 substrates under pressure. This approach completely eliminate strong spectral interference from PET films by measuring the transferred surface layer and greatly improved the signal-to-noise ratio of the surface layer absorption with the Ag island films. The detection limit of PDMS surface layers on PET films was approximately 0.2 nm. The method gave a considerable amount of information about surfaces, such as chemical composition and chemical structure (*EE4*).

Ishino and Ishida propose a nondestructive technique for the measurement of IR spectra of thick, hard, and dark materials by the use of external reflection spectroscopy. The distortion of the absorption bands, which is due to optical effects, can be minimized with the use of parallel polarized light which is incident at Brewster's angle. With this technique, it is impossible to observe overabsorption even if the material has a semiinfinite thickness. Also, this technique can be applied to the study of thin films on polymer surfaces (*EE5*). Ikeda and Uchihara demonstrate that rapid solvent elimination for condensation of sample into a small single residual at room temperature can be accomplished by solvent evaporation on perfluorated polymer film. Spectra of the residual can be recorded by the microscope FT-IR technique. This sample condensation method, namely, the pinpoint condensation technique, provides high sensitivity for IR analysis. The detection limits are at the subnanogram level, and the sensitivity is approximately 1000 times as high, in comparison to the 1-mm-diameter KBr tablet method. This technique can open the possibility of being used in liquid chromatography FT-IR solvent elimination interfacing (*EE6*).

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