

# Gas Chromatography

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This review surveys developments in the field of gas chromatography since publication of the last review in this series (178) and covers the years 1972-73. Gas chromatography continues to be one of the most active areas in analytical chemistry. In the 1973 Directory of Membership of the ACS Division of Analytical Chemistry (356), those listing their research specialty as "gas chromatography" were second in numbers only to those listing the specialty, "general analytical." The most recent report by the American Chemical Society (720) of specialty choices of 84,925 chemists and chemical engineers who reported to the 1970 National Register of Scientific and Technical Personnel shows that 20.9% of analytical chemists list chromatographic analysis as their first specialty choice.

The sale of chromatographic equipment is also an indicator of activity in this field. There are 35 major manufacturers of gas chromatographic equipment with 138 different models currently available (580). We estimate that 2700-2800 articles directly involved with the theory, apparatus, and novel applications of gas chromatography were published during each year in the 1972-73 biennium. This estimate may be compared with a more accurate count of 2665 papers in 1971 and 2555 in 1970. Because of this vast literature, considerable selection was necessary in preparing this review. Technique-centered aspects are mainly considered and most such publications through November 1973 are noted.

## BOOKS AND REVIEWS

Books on gas chromatography published during this biennium include, "Techniques of Combined Gas Chromatography/Mass Spectrometry: Applications in Organic Analysis," by McFadden (577) which, in addition to discussions of GC and MS, contains material on vacuum technology, computer methods, and approximately 15 "mini-papers" on applications; Volume II of "New Developments in Gas Chromatography" edited by Purnell (698); a second edition of Jeffrey and Kipping's text, "Gas Analysis by Gas Chromatography" (426); "Chromatographic Systems; Maintenance and Troubleshooting" by Walker, Jackson, and Maynard (904), covering troubleshooting of both GC and LC equipment; and "Identification of Organic Compounds with the Aid of Gas Chromatography" by Crippen (182) emphasizing the combination of classical qualitative organic functional group analysis with GC retention data and solubility studies for the identification of organic compounds. "An Introduction to Separation Science" is a new monograph by Karger, Snyder, and Horvath (458). Extensive literature references and analytical details for the chromatographic (primary GC, TLC, and paper) determination of the environmental toxicants, carcinogens, mutagens, and teratogens are contained in a recent monograph by Fishbein (260) which forms Volume I of a series on the "Chromatography of Environmental Hazards." An English translation of a Russian monograph entitled, "Gas Chromatographic Analysis of Trace Impurities" by Berezkin and Tatarinskii (75) was just published. Volume VI in the series, "Analytical Methods for Pesticides and Plant Growth Regulators" entitled, "Gas Chromatographic Analysis" by Zweig and Sherma (955) contains 128 individual chapters, each devoted to the analysis of a major insecticide, fungicide, herbicide, plant growth regulator, nematocide, or soil fumigant. "Advances in Chromatography, 1973," the proceedings of the 8th International Symposium held in Toronto, Canada, in April 1973 has appeared (951), and the 66 pa-

pers in this symposium may also be found in the April, May, and June 1973 issues of *Analytical Chemistry*. A Russian Advances in Chromatography series includes reviews on selectivity in adsorption chromatography (482), trace analysis (442), zeolites in gas adsorption chromatography (861), preparative GC (847), and the analysis of gases (421). A number of Russian review monographs were published in 1972 on the subjects: gas chromatography in polymer chemistry (70) listing 826 references; chromatographic applications in catalysis (723) with 629 references; methods for the determination of pesticides in biological materials (935) with a bibliography of 460 references including 117 Russian papers; GC in investigations of natural gases, petroleum, and condensates (495) with 220 references including 148 in Russian, partly from inaccessible sources; and automatic detectors for gases (249) with 143 references, a number from less accessible Russian papers.

The proceedings of the 6th International Symposium on Chromatography and Electrophoresis held in Brussels, September 14-16, 1970 is now available (67) as is an English translation of "Aspects in Gas Chromatography" edited by Struppe (824), the proceedings of the 6th Symposium on Gas Chromatography held in East Berlin in 1968.

A short summary (299) of the Symposium on Chromatography and Computers—An Economical Approach for Every Laboratory, held at the 162nd National Meeting of the American Chemical Society in Washington, D.C., accompanied papers from this symposium published in the December 1971 and January 1972 issues of the *Journal of Chromatographic Science*. Summaries of three informal symposia of the British Gas Chromatography Discussion Group (222, 223, 318) and of the 5th All-Union Seminar on the Theory and Application of Stationary Phases in Gas-Liquid Chromatography (500) have also appeared.

Several monographs were published during the 1972-73 biennium containing useful reviews of GC. These include the gas chromatography of phosphorus compounds (242), of triglycerides (533), and air quality instrumentation (748).

Review papers were published on the determination of thermodynamic functions (499), the application of glass capillary columns in GC (795), applications of capillary columns with special attention to papers of Russian authors (790), relationships between molecular structure and retention indices (842), GC applications in forensic toxicology (181), cannabis constituents in smoke and body fluids (586), local anesthetics and other applications in pharmacy (116), the problems involved in trace analysis in the range  $10^{-2}$  to  $10^{-3}\%$  (74), multistage separation schemes such as parallel and combined columns and backflushing (889), the physical properties and characteristics of porous polymer sorbents including Porapak P, Q, N, R, S, T, Chromosorbs 101, 102, 103, 104, 105, PAR-1, PAR-2, the Czech sorbent Sinachrom, and the Russian sorbent Polisorb (743), methods for multicomponent hydrocarbon analysis (681), steroids (650), applications of GC in Russian space research (106), volatile inorganic compounds (25), the experimental aspects of Molecular Sieves (20), and the status of glass as a GC support material (258), the discussion of which includes the elimination of surface active sites, etching, the application of surface textures and wide-pore diameter glass beads.

The May 1972 special issue of the *Journal of Chromatographic Science* on the Analysis of Drugs of Abuse includes a bibliography of 467 references on the analysis of

drugs compiled by the editorial staff (423) and articles on applications of GC in alcohol analysis (417), hallucinogenic drugs (818), the analysis of narcotic analgesics and amphetamines (622), barbiturates (446), and phenothiazine drugs (158).

The February 1972 and the January 1973 issues of the *Journal of Chromatographic Science* are devoted to a complete listing of over 500 manufacturers of chromatographic instrumentation, accessories, supplies, and services (424, 425).

The 1973 applied review issue of *Analytical Chemistry* cites several hundred references to applications of gas chromatography published during 1971 and 1972 in the fields of air pollution (621), clinical chemistry (305), coatings (834), essential oils and related products (345), food (934), pesticide residues (854), petroleum (480), pharmaceuticals and related drugs (172), high polymers (597), rubber (898), and solid and gaseous fuels (376).

The Preston Technical Abstracts Co. continues its invaluable service to workers in the field of gas chromatography by issuing abstracts of all papers published in the field (690). The abstracts are available in bound booklet form, issued monthly, and carefully referenced to aid in literature searches. Approximately 2400 abstracts are published per year. The thirteenth issue each year is an in-depth subject and author index of the literature abstracted during the year. Both 16-mm and 35-mm microfilms of the abstracts are also available for use with a microfilm reader. A computerized information retrieval system of the gas chromatography literature allows searches of over 26,000 abstracts. Magnetic tapes updated annually are available (690) and are designed for use on an IBM Type 360/DOS computer.

## COLUMNS

**Column Theory.** A great deal of research was published in a continuing effort to better understand the complexities of column behavior. Often this work resulted in the development of new chromatographic techniques, which were reviewed by Zhukhovitskii (947). Impulse-hydrodynamic circulation chromatography (151, 152), diffusion-adsorption analysis (476), chromatography using the method of fixed concentrations (948), flat disk columns (230), derivative gas chromatography (740), and continuous surface chromatography (829) are a few examples. Macnaughton and Rogers (559) applied a principal component analysis to the deconvolution of two or more overlapped peaks. However, high precision GC is a prerequisite in order to reproduce the synchronous timing required.

A mechanism was postulated by Deans (196) for the phenomena observed with trace components when the GC column is overloaded with sample. It was shown that column overloading at the column inlet will cause the sample to act as a stationary phase for components eluting after the major component. Guiochon (352) described the theoretical and experimental considerations in the analysis of low volatility materials. Micro-packed columns of Spherosil in 0.6- to 0.8-mm i.d. columns generated 50,000 theoretical plates at moderate inlet pressures (179). Techniques such as countercurrent continuous GC (26, 27) and frontal GC analysis (712) were well developed in principle and in practice to increase the feed rate and for organic elemental (C, H, N) analysis, respectively. On the other hand, an increasing number of groups sought to compare GC and liquid chromatography with the objective of developing a unified theory (201).

From a study of distribution equilibria in heterogeneous mixtures, Gerritse and Huber (295) measured the adsorption isotherms of two components simultaneously. Liao and Martire (527) developed an alternative approach to treating concurrent solution and adsorption phenomena. Other measurements of physicochemical parameters included the determination of infinite dilution relative volatilities (848) and permeability coefficients by using sorbent heating in a fluid layer (878). A detailed treatment of diffusion in GC was developed from an exponential function utilizing median time and a new parameter describing the concentration vs. time relationship in cases where diffusion is the principal spreading factor (372). A generalized equation was developed to predict the diffu-

sivity of binary systems as a function of pressure and temperature on the basis of diffusion coefficient measurements by GC (402). The effect of natural convection on retention times was noted (12) and a study of columns with flow maldistribution indicated that flow mixing devices or baffles can improve the efficiency (595). Flow patterns in columns were shown by Hawkes (379) to be of interest because they determine the flow rate, the mechanism of mass transfer, and zone dispersion. Thus, extended HETP models were developed, correlated with experimental data, and shown to be superior to the original van Deemter equation (77).

Statistical moments are a sensitive method of peak shape analysis and were used in a number of modeling studies because they have physical significance. A bi-Gaussian distribution served as an empirical model in predicting lower statistical moments and for describing the relationship between impurity fraction and resolution (126). Grubner and Underhill (335) found the first moment to be independent of mass transfer while skew and excess vary as  $1/\sqrt{N}$  and  $1/N$ , respectively, where  $N$  is the number of theoretical plates. The effect of a particle size range on mass transfer was characterized by a moment analysis of elution curves (868). An empirical bi-Gaussian distribution was used to predict lower order moments by Buys and De Clerk (127) for the case of nonlinear elution chromatography. Grushka (336) characterized exponentially modified Gaussian peaks by moment analysis to examine the theoretical behavior of strongly overlapped peaks. In order to measure diffusion and adsorption effects, and variables such as the geometry of the stationary phase or the contribution of a substituent group on the adsorbate from statistical moments, Dwyer (234) used Fourier analysis to deconvolve the column profile from experimental curves.

The significance of interpreting statistical moments has been limited by the accuracy with which they could be measured. Grubner (333) employed inflection points to derive the moments, while Chesler and Cram (149) derived an eight-parameter function for iterative curve fitting by nonlinear residual least squares analysis. Both methods are applicable for asymmetric peaks. Kelly and Harris (472) showed that all information in a chromatogram is available in a section centered at the mean of the peak with a length equal to eight standard deviations of the peak plus the autocorrelation width of the base-line noise by the maximum posterior probability technique. McNair and Cooke (581, 582) pointed out the oversimplification of using Gaussian peaks in their treatment of column overloading by moment analysis.

Instrumental effects contribute to the deterioration of peak shape and should be accounted for in making accurate measurements (640). An in-depth study of the effect of dead volume on efficiency by Maynard and Grushka (572) concluded that the diameter of connecting tubing should always be less than the column diameter. Equations were derived to describe hydrodynamic band broadening (153). Band shape was also described in terms of column processes for frontal analysis (751).

Mobile phase contributions to the plate height showed a nonlinear dependence upon fluid velocity and therefore confirmed "coupling" according to Knox and Saleem (492). At the same time they showed that the stationary phase contribution to plate height obeyed the equation  $H_s = C_s \bar{u}$  and that  $C_s$  does not decrease with retention as much as theory predicts for column capacity ratios exceeding unity. The relationships governing the nonideality of the chromatographic process cause a shift in peak maxima which was explained in terms of the mobile phase velocity (919).

In a GC study of donor-acceptor complexes, Eon, Pommer, and Guiochon (244) calculated equilibrium constants, taking into consideration the activity coefficients and entropic effects resulting from the difference in molecular sizes between an inert solvent and the complexing agent. Their experimental work demonstrated the sensitivity and precision for studying molecular interactions (245). GC was found to be more reliable than either NMR or spectroscopic methods for determining small stability constants of  $\pi$ -complexing in organic solvents (699). The results of the measurement of complex association con-

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stants were interpreted in terms of the electronic and steric effects of substituent groups to give some indication of the type of bonding occurring between substituted alkenes and silver ions (530). Activity coefficients at extreme dilution were measured by Popescu *et al.* (687) by using solvent mixtures as stationary phases. A method was described for the determination of group contributions to activity coefficients through homologous series trends (752). Hussey and Parcher (404) applied a modified Wilson's equation for excess free energy to a number of chromatographic systems and showed that a single limiting activity coefficient may be used to predict the compositional dependence of the activity coefficient. Applicability of Hammett's rule for relating retention time and  $\sigma$  values was found to be poor because only inductive effects have been considered to date (656).

Theoretical analysis of chromatographic microreactor theory showed the possibility of using nonstationary impulse methods for determining adsorption and desorption velocity constants for starting compounds as well as for products from catalytic conversions (931). The interactions between polymers and low molecular weight compounds were evaluated by sorption-desorption thermodynamics (877), and Raoult's law activity coefficients were obtained on polymer systems such as bacteriostatic agents (877). Elution profiles with excessive skewness enabled Schep and De Clerk (753) to obtain polymer reorganization rates by GC. The importance and prospects of GC in catalytic research were reviewed and special interest was noted in the study of kinetics of heterogeneous catalytic reactions (722). A comprehensive review of the applications of GC to catalytic studies was made by Choudhary and Doraiswamy (154), and the chromatographic behavior of fixed bed reactors was predicted when axial dispersion, particle to fluid diffusion, interparticle diffusion, adsorption, and surface reactions may all affect the results (832).

A statistical study of GC operating conditions reported in literature publications shows an increasing tendency toward GSC, separations of multicomponent mixtures, decreased analysis time, and high sensitivity detectors (72). Studies involving the factors affecting separation conditions on HETP showed that while the square of the amount of liquid phase is always significant, an interaction term between the sample size and the liquid phase loading was statistically significant only some of the time (78). Basic equations relating the separation time to operational parameters were derived by Smuts and Pretorius (802). Deans and Scott (200) described the theory and

practice of adjusting the contributions of columns to mixed systems in terms of pressure control. In cases where two columns containing different stationary phases were connected in series and used as one column, artificial change in the gas-velocity ratio in the columns caused a change in the relative retention times of the eluted compounds.

Guiochon and Jacob (354) explicated the theory of separation of mixtures using separation factors to investigate mass exchange between mobile and stationary phase, transfer of mobile phase, influence of discontinuity, diffusion, temperature, and the evaluation and selection of constituent factors in determining the physical model of GC. The theory of zone spreading under conditions of nonequilibrium in helically coiled columns was not possible directly because of angle dependent velocity profiles found in the columns (857). Bogdanchenkov and Vigdergauz (95) proposed that a correction coefficient equal to the square root of HETP be used for characterizing column efficiency with multicomponent separations. In addition to the improvement in separation under conditions of overloading by multiple thermoadsorption concentration (940), theoretical and experimental studies were published on exponentially loaded packed columns (118) and the prediction of the average film thickness in capillary columns (353). The yield in chromatographic separations was obtained from the difference between the sum-abundance values of the two fraction limit volumes involved (913).

Two aspects of overlapping peaks were investigated theoretically by Grushka (337). The peak shape was analyzed with statistical moments in order to discern double peaks and then the resolution was optimized by a length-temperature normalization. Buys and De Clerk (128) used expressions for the statistical moments in nonlinear chromatography to formulate an expression for the resolution function in terms of column parameters. A graphical analysis of three overlapping peaks was carried out by the peak height ratio method (616), and a peak-to-valley ratio method was proposed which was stated to be applicable even to severely tailing peaks (183). Strongly overlapped peaks ( $R < 0.5$ ) were recognized with the aid of a slope analysis employing the second derivative extreme ratios (342). Swingle and Rogers (837) predicted the resolution of pairs of *n*-alkane homologs by assuming that the HETP and resolution were relatively independent of carbon number at a constant capacity ratio.

The validity of maximizing resolution in a given analy-

sis time by length-temperature time normalization was verified by Grushka and Lo (341). The capacity ratio and amount of stationary phase was also optimized in time normalization chromatography (338, 339). McCoy and Cram (575) extended the time normalization theory of GC to predict optimum separation conditions and applied these equations to the high speed separation of metal beta-diketonates. Nomographs were computer-generated for optimizing chromatographic separation schemes of multicomponent samples (569) and a new criterion for the separation of multicomponent mixtures was proposed (932). The latter method was based on the resolution coefficient of all component pairs in the mixture as well as the total analysis time. In this manner, the optimum operating method can be selected with respect to complex column efficiency and analysis time relationships.

**Liquid Phases.** The effect of the molecular weight of the liquid phase on the retention index and other solution parameters at infinite dilution was investigated by Fritz and Kovats (279). Surface tension and its relationship to polarity was also studied (682). A review covering the development and application of Rohrschneider constants for rapidly characterizing liquid phases and correlating molecular structure with retention behavior has appeared (361). An equation was derived (369) extending the earlier work of Rohrschneider and allowing the prediction of the specific retention volume of *n*-octane on 65 liquid phases with an average error of only 9%.

A number of papers were published on the characterization and selection of preferred liquid phases (383, 469, 517, 566, 725, 774, 912). Keller (469) summarized the various recommendations and the responses to them for standardizing and limiting the number of liquid phases in use in GC. Isenhour and coworkers (517) suggested a set of 12 preferred liquid phases and a nearest-neighbor substitution for most of the phases in current use. Mann and Preston (566) propose that the initial preferred liquid phases for determining retention data consist of Apiezon L, methyl silicone polymers, methylphenyl silicone polymers, trifluoropropyl silicone polymers, polyethylene glycol 20M, diethylene glycol succinate polyester, or the equivalent of these materials. A relationship between frequency shifts in the infrared spectrum of a solute dissolved in a liquid phase and its retention on the liquid phase in gas chromatography was reported (236). Eighty-one liquid phases were characterized by the partition coefficients of selected solutes (octane, toluene, ethanol, methyl ethyl ketone, dioxane and nitromethane) by Rohrschneider (724), and the suggestion was made that retention of other substances should be predictable from the partition coefficients of these standard substances. Two other significant papers involving polarity and the chromatographic characterization of liquid phases and solutes for column selection and identification were authored by Rohrschneider (725) and by Novak *et al.* (649). The latter considered a definition of polarity said to be based unequivocally on physical principles, and recommended the difference between values of  $(RT/0.434) \log V_{g,p}^{\circ}$  for butyl acetate and amyl acetate, or two other successive acetate homologs, to be used to determine the polarity of the liquid phases, the polarity being expressed in units of calories/mole  $-\text{CH}_2-$  group.  $p^{\circ}$  in this expression is the saturation vapor pressure, and the other terms have their usual significance. The McReynolds system for characterization of stationary phase polarity was considered by Takacs and coworkers (840). Molecular interactions, polarity and other selectivity parameters, affecting solute retention as described by test solutes, or functional probes, were reviewed by Keller (468). A piezo-quartz microbalance was used to determine the volatility of tricosane, squalane, dotriacontane, and tetratriacontane liquid phases at temperatures up to 400 °K (413). A pre-column containing the same liquid phase as the analytical column, when maintained at the analytical column temperature, effectively saturates the carrier gas with the stationary phase and prevents change in liquid phase weight on the analytical column (484). Oxidation of PEG 4000 and squalane occurs when air is used as the carrier gas (28).

Several studies were made on electron donor-acceptor complexes formed between the solute and the liquid phase such as heterocyclic derivatives with dibutyltetrachloro-

phthalate (245) and aromatic amines with 2,4,7-trinitrofluorene dissolved in a stationary phase (186, 187).

A table of equivalent liquid phase loadings for Chromosorb P, W, and G was prepared (233) considering differences in packing density of the various supports. Nikelly (639) studied the advantages of dual-loaded columns in which the inlet end of the column is more heavily loaded with liquid phase than the outlet end and concluded that such columns were superior in trace analysis where the volume of injected sample is large.

A number of authors reported studies on chemically bonded stationary phases in gas chromatography (38, 284, 343, 457, 536, 537). Locke (536, 537) reviewed the commercially available silicas useful in making chemically bonded phases, the principal methods now used for preparing bonded phases, and the retention mechanism of these substrates. A permanently fixed, or bonded, cross-linked polymer may be formed within the surface pore structure of the support by first coating the support with a solution of a monomer (such as divinylbenzene, ethylvinylbenzene, or styrene) and an initiator followed by gentle heating to product the porous copolymer on the surface of the support (284). Porous silica, when condensed with primary alcohols to form  $\equiv\text{SiOR}$  type bonded phases deactivates the surface but a significant amount of activation remains from unreacted silanol groups (457). Grushka and Scott (343) prepared a permanently bound stationary phase of polyglycine peptide bound to (a) resin coated glass beads, (b) Porasil C, and (c) Corasil II for use in the liquid chromatographic separation of certain amino acids with water as the mobile phase. Poly(ester-acetals), such as those obtained by co-polymerization of methyl azelaaldehyde pentaerythritol acetal (a spiro acetal diester), dimethyl 1,3-cyclohexanedicarboxylate and diethylene glycol, cross-linked and bonded to silanol groups of an acidic siliceous support were recommended as polar stationary phases for use from  $-60$  to  $200$  °C (632). The *in situ* polymerization of silicone liquid phases from monomers of octadecyltrichlorosilane, dimethyldichlorosilane, and phenylmethyldichlorosilane on Chromosorb G and W supports was also reported (375).

Other materials evaluated as liquid phases for gas chromatography include a series of tetrachloroterephthaloyl oligomers (514), a variety of substituted polysiloxanes (32, 360, 939), bis-(2-ethylhexyl)- and di-*n*-butyl-tetrachlorophthalates in the separation of  $\text{C}_7$  to  $\text{C}_{10}$  olefins (739), a series of 3-alkoxypropionitriles (563), silanized and unsilanized Dexsil 400-GC (686) and Dexsil 300-GC, 700-8-73, and 700-8-95c for high temperature capillary column work (651), and *n*-heptyl hypophosphorous acid for the separation of high boiling paraffins, aromatics, and nitriles (16). The tetragonal clathrate,  $[\text{Ni}(4\text{-MePy})_4(\text{NCS})_2] \cdot 0.6$  hydroquinone, was found to be better than any other column packing material for the separation of *o*, *m*, and *p*-xylene at temperatures less than  $90$  °C and gave an elution sequence opposite to that obtained on other packings (838). Amines, Lewis bases, and phosphites are readily separated using nickel(II) dithiophosphinato complexes as the stationary phase (205).

Dilute aqueous solutions of free carboxylic acids may be rapidly determined with columns packed with 10% SP-1200 plus 1%  $\text{H}_3\text{PO}_4$  or in a glass column packed with the porous polymer, Chromosorb 101 (662). The best column for resolving phenols is said to be a capillary column coated with a mixture of 90% dodecyl phthalate plus 10%  $\text{H}_3\text{PO}_4$  (216). Nujol was recommended for the analysis of bis(trimethylsilyl)acetamide (BSA), hexamethyldisiloxane (HMSO), and trimethylsilylacetylacetamide (MSA) mixtures (800). A brown, viscous oil, believed to be 4-dodecylidethylenetriamine succinamide (DDTS), is effective in the analysis of amines and aminoesters (429). Unreacted vinyl groups in Chromosorb 102 are the active sites giving rise to the extreme tailing observed with amine peaks (385).

Several authors reported the special selectivity of organometallic liquid phases. Rhodium(II) benzoate in squalane is selective for compounds such as ethers, ketones, esters, and olefins (764). Dicarbonyl rhodium  $\beta$ -diketonates dissolved in squalane are said to show marked selectivity in their interaction with olefins (298). The silver-olefin charge transfer complexes are well known (349). Cobalt phthalocyanine coated on graphitized carbon black

is a specific adsorbent for aromatic compounds (887). Alkali metal salts of benzenesulfonic acid are claimed to affect retention through hydrogen bonding, clathration, and interaction with the metal ion (80). Russian workers also used alkali metal (509) and alkaline earth (782) halides and nitrates as column packing materials.

Interest in the application of liquid crystals as liquid phases continues. The 4,4'-di-*n*-alkoxyazoxybenzene homologs (23); *p,p'*-azoxyphenetol and its eutectic mixture with *p,p'*-azoxyanisole, and *p*-heptoxybenzoic acid (888, 893); hexyloxybenzoic acid and two azo compounds (466); 4-butyl-*N*-(4-ethoxybenzylidene)aniline (504); *p*-phenylene-bis-3-*n*-heptyloxybenzoate, *p*-(*p*-ethoxyphenylazo)phenyl crotonate, and cholesteryl cinnamate (688); and seven derivatives of 2,5-diphenylpyrimidine (505) are among the liquid crystal phases studied in some detail. The liquid crystalline phase, 4,4'-dimethoxyazoxybenzene, was used to coat a 300-ft stainless steel capillary column allowing almost complete separation of *m*- and *p*-xylene (344). Serpinet and coworkers (776, 777) noted that normal paraffins applied in a light load to kieselguhr gives rise to two rapid transitions in the retention of solutes, one of which is situated below the melting point, but this phenomena is absent when silanized kieselguhr is used. Rogers and coworkers (849) found that an electric field applied across a capillary column coated with a cinnamate ester liquid crystal phase produced a more symmetrically shaped peak with increased retention.

The application of mixed stationary phases for resolving complex mixtures of compounds continues to be studied. A thorough study has been reported in a series of papers in Japanese by Mitooka (598-608). A linear relationship was found between the retention indices of solutes and the weight fraction of the mixed phase provided the difference in relative polarities is less than 30 (600). Some deviation from linearity occurred with larger differences in relative polarity, and this was studied thermodynamically (603). Other studies include the effect of mixed packings on column efficiency (602), the superiority of columns with mixed packing compared with columns with mixed liquid phases (605) and coupled columns (683), and optimum packing composition determined with a digital computer (606).

The ureide of *L*-valine isopropyl ester has been used in the separation of enantiomeric secondary amine derivatives, and the GC behavior of a series of systematically substituted, optically active, peptide derivatives were evaluated as liquid phases (171). *N*-Trifluoroacetyl-*L*-norvalyl-*L*-norvaline cyclohexyl ester is useful as a liquid phase for the enantiomeric separation of *N*-trifluoroacetyl-*D,L*-amino acid isopropyl esters (671). *N*-caproyl-*L*-valine-*n*-hexylamide and *N*-trifluoroacetyl-*L*-phenylalanyl-*L*-leucine cyclohexyl esters (332), *N*-trifluoroacetyl-*L*- $\alpha$ -amino-*n*-butyryl-*L*- $\alpha$ -amino-*n*-butyric acid cyclohexyl ester (672) and *N*-trifluoroacetyl-*L*-alanyl-*L*-alanine cyclohexyl ester (669), and optically active dipeptide phases also proved useful for the separation of derivatives of amino acid enantiomers (670).

The chemistry of polyethylene glycols was described by workers at Union Carbide Corp (678). When Carbowax 20M is coated on Chromosorb W, heat-treated at 280°, and exhaustively extracted, the remaining support performs chromatographically as a highly efficient, well-deactivated GLC packing (39). The physical properties of commercial samples of Apiezon L, M, and N were also reported (630) and evaluated as liquid phases (135) at temperatures up to 300 °C. A new Russian stationary phase, VNII NP-300A, substitute for Apiezon L, M, or N, was compared with these liquid phases and with silicone FM-1322 (629). The chemistry of methylsilicones (859), other silicones (167), and polyester liquid phases (19) including the synthesis, the purification, and the chemical and physical properties has been reviewed.

**Solid Supports.** The factors contributing to the preparation of an efficient column and the structure and surface properties of chromatographic supports were reviewed by Ottenstein (661) and Rotsche (731), who also described various methods of deactivation. Reactivity of solid supports was found with materials such as Chromosorb 102 which depolymerizes in the presence of oxygen at high temperatures (634). Sorptive interactions of water-

modified soil, silica, and Chromosorb W were studied by Okamura and Sawyer (655), who found that GC could thereby provide new insights into the nature of water binding to these materials.

An evaluation of 25 different supports reported their effects on the separation of fatty acid methyl esters (508). Polish workers proposed a mechanism for support surface adsorption in GC on the basis of adsorption isotherms (900). They also used a method for the determination of the adsorption potential to estimate the energy heterogeneity of adsorbents and support surfaces (901). A unique GC method was developed for characterizing partial isotherms of multiphase sorbent and adsorption isotherms on liquid stationary phase-solid adsorbent surfaces (71). An approximation of the contribution of adsorption effects to the retention volume in the presence of concurrent solution and adsorption phenomena showed the effects of support silanization (826). Supryniewicz and coworkers (828) also demonstrated the influence of silanization on the mechanism of support-surface adsorption. An improved vacuum silanization treatment was reported which reduced the effect of traces of moisture on the retention indices (403, 786). Modification of solid support surfaces with surface active agents lead to marked decreases in retention volumes due to reduced absorption on interfaces (403, 555). Chromatographic supports were graded according to particle size in gas and liquid streams (851), and a device for preparing column packings by spraying solid supports with clouds or fogs of solutions was described (53).

Filbert (258) described glass bead technology from the standpoint of the elimination of surface active sites, etching, methods for improving efficiency, and the use of wide pore diameter glass beads which are effective because of their uniformity of pore structure. Zoccolillo and Liberti (953) deposited a thin layer of carbon on glass beads by pyrolyzing CH<sub>2</sub>Cl<sub>2</sub> at 550 °C, and found them to be superior to untreated glass beads. Both Russian clay (475), and an Indian attapulgite (56% silica) (646) were found to be satisfactory for nonpolar separations of light hydrocarbons. Special precautions were reported for packing highly uniform columns of Chromosorb T (Teflon 6) (4).

Schwartz *et al.* established the GC behavior of dendritic salts as solid supports (768) and porous attrition-resistant spherical supports for high speed analysis (767). Poly(*cis*-butadiene) was coated onto a conventional support to act as a controlled surface adsorbent (14). Similarly, terephthalic acid was prepared as a solid support for trace analysis of SO<sub>2</sub> and H<sub>2</sub>S in H<sub>2</sub> (607). New solid supports were reported for the separation of aliphatic alcohols (635) and Al and Sn supports gave the best separation of aromatic amines among the metals investigated (308). Eleven different diatomite supports were compared for the analysis of phenols (825).

**Adsorption Columns.** In his treatment of adsorption phenomena in gas solid chromatography (GSC), Berezkin (69) reviewed topics such as the contribution of interfacial adsorption to retention, Gibbs adsorption coefficients, and thermodynamic properties of adsorbent surfaces. Adsorption in GLC was also treated in terms of its effect on retention (292). However, Eon, Chatterjee, and Karger (243) pointed out that liquid-solid adsorption is not an important contributor to GLC.

The molecular theory of adsorption in chromatographic separations was interpreted in an extensive review by Kiselev (482). Special attention was given to selectivity, as interpreted on a molecular basis.

Other thermodynamic studies determined Henry's law constants, isosteric enthalpies, potential energies of adsorption, surface areas of adsorbents (102), differential heat of adsorption for cases where the gas phase is not ideal (91), and the internal diffusion activation energy (879). MacDonald and Habgood (557) determined internal diffusivities in zeolite crystals by comparing the peak spreading in two different carrier gases. The observation that the log of the surface area of adsorbents varies linearly with changes in retention times for homologous series led to the development of a simple and rapid GSC method for the determination of surface area (296). An experimental verification of Haarhoff's (*Anal. Chem.*, 38, 573,

1966) treatment of adsorption, taking into account nonlinear isotherms, showed the theoretical solution to be applicable to parabolic isotherms (438). The temperature dependence of selectivity coefficients in adsorption were measured for ortho- and para-hydrogen and correlated with quantum theory (87). Distribution coefficients were measured by GSC and compared to static measurement methods (190). Equations were derived to express the effect of adsorption of carrier gas in GSC in order to predict the improvement in peak symmetry, reduction in retention volume, and the independence of relative retention on the type of carrier gas when an inert gas is replaced with an adsorptive gas as carrier (747). A new method for evaluating the third virial coefficient in physical adsorption by GC was described (736). It was then shown that the surface area of adsorbents could be obtained from the third gas solid virial coefficient (737).

Nonspecific interactions on salt-modified aluminas and silicas confirmed the validity of the King-Benzon theory (*Anal. Chem.* 38, 261, 1966) (291). Isbell and Sawyer (410) used salt-modified silica in their studies of adsorption thermodynamics of functional groups on substituted hydrocarbons. Sodium chloride impregnated, sodium hydroxide modified alumina was characterized by the separation of polycyclic aromatic hydrocarbons (883). Proton magnetic resonance studies of salts on chromatographic gels indicated that the mechanism for separation of salts was by selective adsorption (679). Experimental problems associated with the use of inorganic salts as column materials were discussed (509). High temperature treatment of BaSO<sub>4</sub> improved its selectivity for separations of aromatic hydrocarbons, alkylaromates, polyphenyls, and condensed nuclei hydrocarbons (68). Hydrocarbon separations of the chlorides of V(II), Mn(II), and Co(II) were studied to ascertain the effect of the differences in the availability of their "3d" electrons (329).

Kiselev (481) outlined basic methods for developing and improving adsorbents for GSC to ensure their high geometrical and chemical homogeneity, thermal stability, and controllable specificity. The selectivity and efficiency of clay minerals with a porous lattice, such as sepiolite and attapulgite, was found to be dependent upon differences in surface geometry and homogeneity (564, 565). Deactivation of a wide pore silica gel was reported by calcination, depositing a nonextractable polymer layer, and by adding small amounts of a polar compound to the carrier gas (43). Modification with steam or alcohols resulted in an increased selectivity as the length of the alkyl chain in the alcohol molecule increased (902). The differential heat of adsorption, specific surface area, and mean pore radius were calculated from the adsorption isotherms of these modified silicas, and showed evidence of water on the esterified silica gel of a polymolecular nature (899). A systematic study of the silica adsorbent, "Spherosil," indicated that the activation temperature, specific surface area, porosity, and sample size were instrumental in affecting the retention, resolution, and other chromatographic properties (351). Because of the hydroxyl groups on its surface, it interacts specifically with molecules that contain a dipole or pi electrons (255). Criteria for selecting the optimum stationary phase thickness for modification of Spherosil were discussed (350).

New adsorbent surfaces can be produced by coating graphitized carbon black with various phthalocyanines and thereby new tailored selective adsorbents (886). By modification of the carbon black surface by paraffins (776) and acetic acid and propylamine (214), even hydrogen bonding compounds were eluted (213). General considerations for the preparation of surface-layer sorbents, their chromatographic properties, and applications were reviewed (73). These materials were characterized and shown to have little dependence of HETP on the carrier gas velocity or on the grain size of the solidsupport.

A new 0.5-nm Molecular Sieve was described for the separation of CO<sub>2</sub> and N<sub>2</sub>O (573). Separation factors of binary mixtures on zeolites were discussed in terms of the presence of pi electrons (117). The role of cations and anions in adsorption separations, both by zeolites (839) and by alkali metal halide salts (541), was investigated with respect to selectivity. Positive changes in the specific retention volumes of some aromatic hydrocarbons were

noted as the Na<sup>+</sup> content on zeolite and the pretreatment temperature were increased (571). Introduction of water into the system caused a reversible change in retention volumes. Other ion exchange forms of zeolites were studied (96), and a review of zeolite adsorption separations appeared in Russian (861).

"Synachrom," a new macroporous styrene-divinylbenzene copolymer, was reported to be stable to 340 °C and comparable in efficiency to other porous polymer column packings (231). Two polyimide-based polymer sorbents were evaluated and found to be highly specific because of the presence of imide and carbonyl functional groups on the surface (742). Porous polyacrylates were characterized by a high nonspecific interaction energy with aromatic hydrocarbons and are specific for molecules with high dipole moments (483). The Russian copolymer "Polysorb" was used for the analysis of glycols and glycerol in the presence of water (865). Other new porous polymers containing phosphorous and nitrogen showed very specific retention behavior (665).

Sakodinsky and Panina (743) reviewed the properties, chromatographic characteristics, and applications of porous polymer sorbents. The lot-to-lot reproducibility and the effect of different methods of conditioning for Porapak were established on the basis of reproducibility of retention data and separation factors (227). Gough and Simpson (316) measured the reliability of porous polymer beads for water-alcohol analysis and attributed variations in the results to preferential losses by adsorption of water in the columns.

A brief note described a method to rectify the variability in performance of Porapak Q in the determination of acetic acid in water (313). Similar problems were corrected by the addition of formic acid vapor for the analysis of free fatty acids without altering the retention times or column life (3). Abnormalities in the chromatographic behavior of isometric compounds (225), methyl branched ketones, and fatty acids (2) were detected in certain Porapak materials (225), and the oxidation of porous polymers by samples containing oxygen was noted (806). The use of polar Porapak was found to be critical for the resolution of polar mixtures, such as formaldehyde/H<sub>2</sub>O (869), and Porapak Q was evaluated at cryogenic temperatures for the separation of <sup>3</sup>He, Ne, and <sup>2</sup>H in <sup>4</sup>He (909). The versatility of the Porapak columns was demonstrated by Deans *et al.* (199), in their separation of complex gas mixtures with a single injection by employing column switching. Various fluorinated porous polymer sorbents were significantly effected by samples such as UF<sub>6</sub> and other corrosive volatile compounds (36).

Extensive application of GSC has been made to all of the adsorbents described here. Continuous chromatography of hydrocarbon gases also used adsorbent beds (51). Bruner and coworkers (112) separated deuterated and tritiated propanes and butanes in columns packed with carbon black which had been treated with 0.2% squalane. Sulfur-containing atmospheric pollutants were determined in the ppb range with carbon black modified with H<sub>3</sub>PO<sub>4</sub> and Dexsil (115). Polynuclear aromatic hydrocarbons were separated on carbon black (280) and on carbon black deposited on Chromosorb W (281). The graphitized carbon black adsorbent columns were also efficiently used for GC-MS of essential oils and food extracts (114).

Deactivated or surface modified alumina and silica gel adsorbents were applied to the separations of hydrocarbons (633), chlorinated hydrocarbons (574), and PCB's (689). In these studies, it was noted that the most active sites on alumina columns, even after deactivation, were the aluminum atoms. An intercomparison of silica gel, charcoal, and Molecular Sieve columns was made for the analysis of smoke gases (785).

Molecular Sieve chromatography may be applied to organic and inorganic molecules in both aqueous and non-aqueous systems and a compendium of their commercial sources was given in a review (20). Several commercial Molecular Sieves were compared for hydrocarbon separations (331). Separations of acetylenes and dienes in propylene (945), furnace combustion gases (98), isomeric triethylbenzenes (627), and cyclic hydrocarbons (287, 680) illustrated their capacity for handling a large sample molecular weight range.

Adsorption gas chromatography of *n*-alkanes (644) and high boiling hydrocarbons (645) was facilitated by using steam as the carrier gas. Steam containing formic acid or hydrazine hydrate was used for the separation of organic acids and amines on diatomaceous firebrick, porous glass beads, and other untreated adsorbents (643). Even preparative scale GSC columns were developed for a wide range of adsorbents, although the peak symmetry appeared to be affected by the carrier gas flow rate (942).

**Open Tubular Columns.** A review of 208 papers on applications of capillary GC for the analysis of multicomponent mixtures has appeared in the Russian literature (790). Retention indices measured on packed and capillary columns were shown to be identical (789). Equations for the prediction of the average film thickness in capillary columns prepared by the dynamic method were described and evaluated by Guiochon (353). Grushka and Guiochon (340) have resolved differences reported in their earlier publications regarding the optimum value of the capacity ratio in time normalization chromatography.

The use of glass open tubular columns received considerable attention during this biennium. A comprehensive review in Hungarian cites 155 references to the literature on glass capillary columns, their preparation and applications (795). On-column injection is recommended by Verzele (885) with wider glass open tubular columns since (i) there is practically no loss in efficiency, (ii) quantitative results are more reliable than when a splitter is used, (iii) samples can be relatively large, (iv) the glass surface is less reactive toward thermally labile materials. Equipment and methods for direct injection into capillary columns were also described by Grob and Grob (328) and applied for the analysis of tobacco smoke. The effect on the thickness and quality of the liquid phase film deposited, of the viscosity of the solvent, and the surface tension of the liquid phase, the solvent, and the glass surface was studied (631, 850). The efficiency and wettability of borosilicate glass for polar liquid phases improves in the order: untreated glass, HF-treated, alkali-treated, chromic acid-treated, and graphitized (290). Modifying the inner surface of glass tubes used in the preparation of capillaries and improved static coating techniques are proposed by Verzele and coworkers (99). An apparatus was developed for dynamic coating of open tubular columns with a constant meniscus velocity allowing the coating of columns of all lengths and diameters with solutions of wide viscosity range (873). Also described was an apparatus for the reproducible coating of liquid phases on glass capillary columns either in the pure form or in admixture with fine particles of solid support with simultaneous silylation of the liquid phase and column inner surface (406). Capillary columns silanized with dimethyldichlorosilane prior to coating with liquid phase are said to form a film on the surface that does not break up into microdroplets (294). Compounds with molecular weights as high as 800, including cholesterol esters, were analyzed at temperatures up to 350 °C using the carborane liquid phases, Dexsil 300GC, 700-8-73, and 700-8-95C (651).

Porous-layer open tubular (PLOT) GC columns may be prepared by passing a plug of a solution of the liquid phase and a suspension of a solid support in a volatile solvent through a coiled capillary tubing (637). Dynamically coated PLOT columns can be made nearly equivalent in efficiency and capacity to those made by the static method (638). Silanox 101 (Cabot Corp.), a colloidal dispersion of a hydrophobic silica with particle diameter of  $7 \times 10^{-7}$  cm, was recommended for the preparation of PLOT columns (93). A PLOT column containing cobalt phthalocyanine on Sterling FTG carbon black separated aromatic amines, pyridine, picolines, and lutidines when a hydrogen and ammonia mixture was used as carrier gas (271).

Mon (613) recommended a cleaning procedure for open tubular columns which consists of washing solutions used in the order: chloroform, acetone, water, concentrated nitric acid, potassium hydroxide in 1:1 methanol-water mixture, water, concentrated nitric acid, water, concentrated ammonium hydroxide, water, acetone, and chloroform. A U.S. patent has been issued on a spiral capillary column consisting of a pair of separable surfaces which define the capillary passage and which may easily be disengaged to permit recoating of the liquid phase on its

grooved surface (745). A frame was developed for capillary columns which will accommodate 1200 ft of 0.0625-in. o.d. tubing in 28 tiers so as to minimize thermal gradients (906). An all-glass solid sampling device, capable of being connected to any chromatograph, is designed for the analysis of trace quantities of high-boiling compounds such as steroids in body fluids (874). A flame photometric detector was coupled to a capillary column and used to detect trace amounts of sulfur compounds (315). By using high-speed differential pumping of the source and analyzer of a mass spectrometer, helium flow rates in the range 0.1 ml/min can be tolerated in the direct coupling of a capillary column gas chromatograph to a mass spectrometer without special interfacing (382), and oxygenated steroids and triterpenoids in the C<sub>27</sub> to C<sub>32</sub> range may be analyzed.

Open tubular columns have been used for the chromatography of human urinary steroids (293, 738), all positional and geometric isomers of C<sub>8</sub> to C<sub>11</sub> *n*-olefins (589), olefins from C<sub>6</sub> to C<sub>14</sub> (808), 69 saturated C<sub>5</sub> to C<sub>8</sub> hydrocarbons from petroleum naphtha (609), and the resin acids in rosin fluxes (374).

## DETECTORS

In a review of the principles of detection in chromatography and the analytical characteristics of detectors, Keller (470) emphasized the importance of technique reproducibility and the use of control samples in analysis. An extensive and detailed review of detectors was also published in Polish (907). The importance of the need for basic studies in detection was shown by Kelley and Harris (471) who statistically characterized detection base-line noise with a power-density spectrum. Peak parameters were established in the frequency domain of the Fourier transform of the chromatogram. Russian workers have proposed a method of determining the absolute and relative sensitivity of chromatographic detectors without measuring peak areas (250, 251).

A number of new concepts and principles have been reported for detection of column effluents. A thermocatalytic detector for detection at low temperatures was constructed by coating alumina and Pt/Pd catalyst layers on a Pt spiral (31). The detector was shown to be stable and moderately sensitive. A diaphragm-type detector was patented for operating at high temperatures (812). A reaction of the column effluent on the surface of a seignettelectric material gave a pyroelectric effect where the pyroelectric charge was proportional to the rate of temperature change (347). As an example, the authors used a lead zirconate-titanate sensing element coated with silver for the detection of diethylether in a He and O<sub>2</sub> mixed carrier gas.

Janghorbani and Freund (419) described the development and characterization of a piezoelectric digital transducer for monitoring sulfur compounds in pulp mill stack effluents. The piezo-quartz microbalance measured the volatility of GC stationary phases by the difference effusion method (413). Another adsorption detector using thermocouples to measure the heat of adsorption (and desorption) in gas and liquid chromatography was investigated in detail by Stafford (819).

Colorimetric tests for organic functional groups at the microgram and submicrogram levels were achieved by Cronin and Gilbert (185). In order to realize this sensitivity, they trapped the GC peaks on short lengths of glass porous layer open tubing containing a layer of activated Al<sub>2</sub>O<sub>3</sub>.

**Thermal Conductivity.** The status and recent developments in thermal conductivity detectors (TCD) as reviewed by Johns and Stapp (432) showed that a minimum detection limit of 1 ppm is possible under ideal conditions. They described significant improvements in the areas of flow geometry, total cell volume, temperature control, sensing elements, and electronics. DeLew (203) stated that a constant current source would extend the linear dynamic range of the TCD by a factor of three. In addition, the detector sensitivity was improved by a factor of ten by use of higher resistance filaments and by reducing the distance from the filament to the cell wall and thereby reducing the cell volume. The influence of temperature on the detector sensitivity under conditions of constant current was studied theoretically by Soloviev

(814). New design considerations to minimize thermal effects were patented (496) as was a system for eliminating the influence of changes in carrier gas flow rate on the detector response (946). The latter employed a sensing element with a negative temperature coefficient of resistance in the reference cell so that the detector operated as a thermoanemometer.

In a novel modification of the TCD, a thermosensitive dielectric wire was positioned such that a higher temperature element was run parallel to it to increase the sensitivity and exclude the influence of gas channel volume on detector response (125). A comparison of the analytical characteristics of a TCD and a TCD with constant filament temperature showed that the constant filament temperature detector had a larger linear dynamic range, higher sensitivity, lower time constant, and comparable lower limit of detection (783, 923). The limitation of sensitivity in the TCD was overcome by using a palladium transmodulator between the column and detector to increase the gas phase concentration of the solute (546). Wade and Cram (897) used a thermistor sensor as a flow-sensing protection system to avoid burning out filament type thermal conductivity detectors. The properties of thermistors as chromatographic detectors were expressed in terms of the mass, specific heat capacity, resistance, and temperature coefficient of the thermistor (532). One study even reported the feasibility of increasing the sensitivity of a thermistor TCD by lowering its block temperature (596). Transistors may also function as the sensor in a TCD at temperatures <200 °C, although they are also flow sensitive (660). The development of an AC or square wave TCD was continued by Ediz, van Swaay, and McBride (237).

Rosie and Barry (727) discussed quantitation of thermal conductivity detectors and tabulated a large number of relative response values. Williams and Carritt (921) generated O<sub>2</sub>, H<sub>2</sub>, or N<sub>2</sub> and H<sub>2</sub> in a coulometer for accurate calibration of thermal conductivity detectors. Various methods of calculating the relative molar response of thermal conductivity detectors has been reported by Monfort (614) and Barry and Rosie (56, 58, 59). From physical properties such as the thermal conductivity coefficients and molecular weights of both the solute and the carrier gas, the response of the TCD can be predicted to approximately 10%. Relative molar response factors were successfully predicted on the basis of the molecular diameter approach for the anomalous response of halogenated compounds (57). It was also suggested that critical volumes could be estimated from a knowledge of relative molar response data (60). Other aspects of quantitation by thermal conductivity such as the variability of response factors for light gases (H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, and He) (787), differential detection systems (97), and pyroelectric effects (348) have been treated.

The gas density balance has realized an enhanced impact through the determination of the molecular weight of chromatographic solutes. The variables which contribute to the response characteristics (195), the accuracy of the detector (894), and calibration techniques (882) have appeared recently.

**Flame Ionization.** Reviews of the characteristics and operational variables of the flame ionization detector (FID) (302), the mechanism of flame ionization, types of flames commonly used, and factors affecting production of ions (94) reflect the continued interest in the development of ionization detectors. Blades (85) published a definitive, although inconclusive, paper on the mechanisms of ion formation with evidence for the CH-O mechanism. He pointed out the conflicting hypotheses of oxidative and reductive determination of equal per carbon response and proposed that this arises in the complete conversion of eluants to CH<sub>3</sub> prior to entering the oxidative zone of the flame.

New features in flame detectors have been reported which claim new electrode configurations for increased linearity, even at very high concentrations (462). Aue and Hill (40-42) reported the use of a hydrogen-rich flame for the detection of tin compounds while repressing the carbon signal. Additional selectivity can be obtained with a dual channel detector for measuring both flame ionization and emission. Aerodynamic disturbances and flame vitia-

tion by recirculating chamber gases in flames at reduced pressures have been avoided by incorporating a flame shroud and an exhaust port of high pneumatic resistance to the detector (551). This improved performance may have a particular impact in atmospheric environmental monitoring. The behavior of a high pressure FID established the detector response dependence on pressure in the 1-100 bar range (92). By separating the jet and collecting electrodes by 4 meters, a restrictor was not required between the column outlet and the detector. A chemical amplifier for the FID in which the combustion of substances over Co<sub>3</sub>O<sub>4</sub> and the interaction of CO<sub>2</sub> and H<sub>2</sub>O with the alcoholates of alkali metals was proposed (741).

Grant and Clarke (319) investigated the quantitative effects of the chromatographic and detector conditions on the analytical precision of the FID and found that the use of a carefully chosen internal standard appears to be essential for achieving high precision. Novak *et al.* (648) showed that the background detector signal (carrier gas plus stationary phase vapor) causes a decrease in the net response of the detector, even when it is operated in the linear range. That the sample size can be increased by at least two orders of magnitude beyond the limit of the linear range of the FID has been shown by Rossiter (730), as the logarithm of the sample size and the peak area are linearly related. Pan (664) took the approach of measuring components in the nonlinear region relative to a minor component and obtained similar results. A steady-state calibration method for flame ionization detectors demonstrated a standard deviation of 1.8% (134). Quantitative studies have shown the relationship of molecular structure and the number of ionizable carbon atoms in substituted picolenes to the relative molar response factors of substituted picolenes (502). Binder and Lindner (82) determined that the low molecular weight esters give the expected relative molar responses for mono- and dicarboxylic acid esters. Other detailed studies have included heteroaromatic compounds (159).

The lack of response or anomalous response of the FID to certain compounds has been studied in detail by many groups. For example, the low response to fluoroalkanes was found to be due to both the initial bond breaking process and in the subsequent conversion of the fragments to the ion precursor, CH (86). Although the response to CF<sub>4</sub> was very low, the response of large carbon number perfluoroalkanes approached that of their hydrocarbon analogs (34). This variation in response values was attributed to the bond strength and molecular structure of the perfluoroalkanes. Karmen and Kelly (461) exchanged fluorine functional groups for chlorine by combustion in a hydrogen flame to HF which passed through CaCl<sub>2</sub> where HCl was released and detected with an enhanced sensitivity in an alkali FID.

Askew (33) detected inert molecular such as CO<sub>2</sub>, SO<sub>2</sub>, N<sub>2</sub>, and He by using an ionizable carrier gas, CF<sub>4</sub>. The dilution of the ionizing carrier gas stream by the inert sample appears to decrease the ion current produced in the FID and thus create negative peaks for the inert samples. The method was shown to be sensitive to 1 × 10<sup>-6</sup> gram mole of N<sub>2</sub> and nonlinear for hydrocarbons. A study of the response of the FID to nitrogenous compounds postulated the ion formation processes for inert compounds such as C<sub>2</sub>N<sub>2</sub>, NH<sub>3</sub>, NO, N<sub>2</sub>O, and PH<sub>3</sub> (84).

As little as 2 × 10<sup>-9</sup> gram of NO<sub>2</sub> was detected by reacting with styrene and using FID to measure the reaction products (334). Schaefer (749) obtained FID responses to O<sub>2</sub> and N<sub>2</sub>O by using a partially premixed flame. The FID can also be used satisfactorily for the analysis of CO or CO<sub>2</sub> by increasing the background current with a trace amount of hydrocarbon or by operating the detector with a hot burner. This response has been explained in terms of temperature changes in the diffusion flame of the FID and their influence on the hydrocarbon response (750). An 18% reduction in the sensitivity was observed for an FID burning in O<sub>2</sub> containing 12.3 mole per cent H<sub>2</sub>O vapor. It was also noted that a signal change of 4.7% was found as the relative humidity in the atmosphere varied from 50 to 100%, which should be noted for environmental monitoring applications (550). Eggertsen and Stross (238) determined organic carbon in water by



vaporization and pyrolysis into a FID.

Alkali flame ionization detectors (AFID) may be readily constructed according to the specifications of Stewart (821) or attached to conventional hydrogen flame ionization detectors (460). Karmen and Haut (460) found that electrical heating of the alkali metal source produced an increase in specificity, presumably because the temperature of the source was less affected by an increase in temperature during the passage of the solvent peak. Reduced background current and noise can be achieved by providing a sealed top for the detector chimney, an exhaust passage for combustion products, and an excess air supply at the base of the chimney (371).

A nitrogen selective thermionic detector described by Riedmann (714) reduced the amount of sample preparation required for biological samples and detected ppm-ppb concentrations of nitrogen-containing compounds. Optimization of the operating conditions of an AFID for the analysis of nitrosoamines gave the highest sensitivity when a Pt-Ir coil was coated with KCl (463). The response of the AFID in both the positive and negative modes to various hetero elements in organic compounds was shown to be proportional to the amount of the hetero atom introduced (37). Other analytical studies reported the selectivity of the detector to fatty nitrogen compounds (418), the response to HMDS derivatives from urine (174), group IV (Si, Sn, Pb) compounds (226), and the determination of amino acids in wort and beer (50).

A chemical ionization detector was reported by Scolnick (770) which can be operated in a specific detection mode for sulfur, nitrogen, and phosphorus in the presence of hydrocarbons. A nonhydrocarbon constituent of the sample was reacted with an alkali metal vapor. A chemi-ionization reaction occurred in the absence of O<sub>2</sub> with certain nonhydrocarbons of the sample and resulting ions were collected in an electrostatic field.

**Electron Capture.** The theory and detection mechanism of the electron capture detector (ECD) were described by Zlatkis and Fenimore (952), and Bothe (101) described experimental aspects of ionization detectors which use radiation sources. Dependence of the ECD response on the pulse interval was based on a kinetic model for the electron attachment processes which assumed that the positive ion concentration was constant (915). Lovelock and Maggs (545) observed that with intensely electron-absorbing substances, more than 90% of the molecules entering the ECD may be ionized. They also noted that when operated coulometrically, thermal electron attachment is a sensitive, accurate, and absolute method of analysis. In his treatment of electron attachment for small molecules, Freeman (276) reported the development and characterization of a detector which was capable of functioning as either a "universal" photoionization detector or as a selective ECD.

Maggs, Joynes, Davies, and Lovelock (560) reported one of the most significant developments in the last biennium for electron capture detectors. They varied the frequency of the applied pulses to the ECD under constant detector current, such that the change in pulse frequency was proportional to the sample concentration. Operated in this manner, the detector behaved as a concentration dependent rather than as a mass sensitive detector. It was found that the detection limit and linear dynamic range varied with the reference current but offered improved performance over the conventional mode of operation. Some of the present complications and controversies with the ECD stem from the fact that a given detector may or may not be flow, concentration, and/or temperature sensitive, depending on the type and amount of solute and the electron concentration (44). In this regard, Chen and Wentworth (146) pointed out that whenever ECD response data are given, the temperature of the detector must be specified and the optimum temperature for the analysis should be determined. The ECD was found to be virtually independent of the temperature and flow rate if the carrier gas did not contain electronegative contaminants such as O<sub>2</sub> (875). The reduced sensitivity and linearity of the detector in the presence of O<sub>2</sub> was explained on the basis of the formation of reactive negative ion-molecular complexes (452). This effect is greatest at lower temperatures and is related to the water vapor concentration in the air.

Comparative studies of new ionization sources indicated that <sup>147</sup>Pm can be electroplated on gold foils and heated to 400 °C without appreciable loss in activity (548). When compared under identical conditions and in the same detector geometry, the background current due to the <sup>63</sup>Ni source was 2.4 times greater than for the <sup>147</sup>Pm source (549). The titrated scandium source can be operated to 325 °C and offers a life expectancy four times longer than conventional detectors (370).

A comprehensive review of EC detectors for the determination of halogenated derivatives of polar compounds was published by Cummins (189). For nitrogen-containing compounds, the ECD sensitivity to nitrobenzene was only 16 ng (393). Therefore derivatives are often used such as perfluorobenzene (610), heptafluorobutyramide, pentafluorobenzamide, and pentafluorobenzylidene (568). Sulfur analyses have been reported to be sensitive to 5 × 10<sup>-3</sup>% SO<sub>2</sub>, 1 × 10<sup>-3</sup>% H<sub>2</sub>S, and 2 × 10<sup>-6</sup>% CS<sub>2</sub> by EC (516), which is also 20 times more sensitive to soluble elemental sulfur than the flame photometric detector (823). Dietz and Cote (215) were able to detect 4 × 10<sup>-13</sup> part by volume of SF<sub>6</sub> with EC by eluting SF<sub>6</sub> before the air peak.

Lukac and Sevcik (553, 554) discussed the detection mechanism of the helium ionization detector using metastable atoms or rare gases, and the response characteristics of the detector. Generally, the helium detector should have a higher sensitivity for substances with a low ionization potential and is stated to be the most sensitive detector for permanent gases, although it is highly sensitive to air contamination of the helium. This was illustrated by Bruner *et al.* (113) in their analysis of 50 ppb of CO in air.

A small volume (~1 ml) flow proportional counter for monitoring <sup>14</sup>C-labeled CO<sub>2</sub> in He carrier gas has been reported with 80% counting efficiency (924). Improved performance of the flow proportional detector for <sup>3</sup>H- and <sup>14</sup>C-labeled compounds was obtained by passing the column effluent through a hydro-cracking tube and counting the <sup>14</sup>CH<sub>4</sub> produced (88). A system for continuous detection by liquid scintillation dissolved radioactive effluents in a stream of scintillation solution and pumped them through a helical cell in the counter (765). Alternately, the effluent was condensed and adsorbed onto small glass beads coated with silicon fluid and eluted into scintillation vials. The overall recovery of radioactive fatty acid methyl esters and TMS derivatives of glycerol and glucose was greater than 90% (908).

**Mass Spectrometry.** The basic principles, modes of operation (766, 820), methods of sampling and interfacing (261), computerization (450), and relative merits of GC-MS interfacing have appeared in a number of reviews. Many authors have emphasized direct coupling of the GC and MS for total effluent analysis. For example, Schulze and Kaiser (762) detected quantities as small as 10<sup>-10</sup> gram from open tubular columns with flow rates up to 10 ml/min without loss in spectral resolution.

Interesting differences in the direct inlet and GC inlet mass spectra have been noted (206). Direct coupling with a cold trap offers the advantages of a high enrichment factor and successive collection of several fractions from a single run (523).

A 15-μl porous silver membrane separator used with open tubular columns operating in the range of 2-7 cm<sup>3</sup>/min gave yields of 25-60% and enrichments from 2-10 (525). Comparison of the GC peak widths from the FID and total ion monitor showed no detectable peak spreading in the molecular separator. Recent data showed that the conductance of the membrane and pore volume are the most important design parameters of the porous silver membrane separators and that a wide range of flow rates can be handled (320). For low background spectra, sliding valves of gold-plated tungsten were developed (512).

Work in chemical ionization (CI) GC-MS has just begun to appear with the advent of commercial instrumentation. Arsenault, Dolhun, and Biemann (30) described an instrument with an electron impact (EI) and chemical ionization source such that both spectra can be obtained from a single injection. The system permits rapid and repeated shifting from one mode to the other without interrupting the GC separation and without a loss in sensitivity. Modifications of a high resolution mass spectrometer for both EI and CI studies (937) and details

of the CI technique for GC-MS have been reported (756). Chemical ionization GC-MS was used to identify noncannabinoid phenols in marijuana smoke (256).

Samuelsson, Hamberg, and Sweeley (744) found the addition of deuterium to the carrier gas enabled them to quantitatively measure prostaglandin by isotope ratios at the nanogram level. A novel means of elemental analyses of sub-microgram quantities of GC effluents employed a combined MID-peak matching technique (364). An excellent review of MID with GC-MS in biological research emphasized the application of MID as a specific GC detector (314). The picomole sensitivity of the technique is indicative of its applications (501).

To realize the full power of coupling GC and MS, new data systems were implemented, generally with dedicated computers and large bulk storage facilities (109, 144, 799, 880). There was a noticeable lack of new algorithms for data handling, peak sensing, etc. The real time selection and correction of mass spectra is one exception (384).

For GC-MS identification of ketones, intensity ratios,  $I_o/I_\Sigma$ , have been used where  $I_o$  is the sum of the line intensities for oxygen containing ions having mass units of 31, 45, 59, etc. and  $I_\Sigma$  is the sum of all line intensities (701). Mass ratios of  $I_{39}/I_{41}$  and  $I_{41}/I_{43}$  were evaluated for identification of homologous series (700).

The principal applications of GC-MS have included clinical biochemistry where it has been applied to serum, urine, and biopsy materials (427), the study of human metabolic profiles (397), the analysis of steroids (709), and environmental samples (286) such as organomercury compounds (63), lead (24), polycyclic aromatic hydrocarbons of air-borne pollutants (515), and aromatic hydrocarbons in river water (390).

One of the most exciting new developments was the implementation of a Plasma Chromatograph reactant ion chamber with a quadrupole mass spectrometer. Horning *et al.* (398) reported picogram sensitivities with the atmospheric pressure ionization source. Reactant ions reacted with biological compounds through charge transfer or proton transfer, and both positive and negative ions were seen.

The first coupling of a gas chromatograph and Plasma Chromatograph (PC) was reported by Cram and Chesler (177). They discussed the GC-PC interfacing in detail, showed the analogy of GC-PC to GC-MS for identification, and compared the sensitivity of a series of Freons to the FID. Karasek and Keller (454) used the same system for the detection of musk ambrette but their sensitivity was severely limited by column bleed. The validity of ion mass assignments for a series of polynuclear hydrocarbons based on mobility measurements in PC reported the effect of substituted functional groups and showed that mobilities may be used as identification aids in GC (325).

Basic patents in PC were issued to Cohen and coworkers covering ion formation and ion drift (164), the enhancement of sensitivity of ion measurements in a drift cell (166), the methods and apparatus for resolving ion molecules (133), a high temperature method (165), and means of improved signal to noise ratios (916). The response of the PC to alcohols and PCB's showed the specificity of the technique through the availability of both positive and negative ion spectra (449, 451, 453). The sensitivity of the PC appears to be at least comparable to that of the ECD; further, the PC promises to be a powerful tool in understanding the behavior of the ECD (456).

**Spectroscopic.** Selucky's (772) review of specific chromatographic detectors described various designs and modifications for making the flame photometric detector (FPD) specific for sulfur, phosphorus, or hydrocarbons. A more specific treatment of the FPD for continuous  $\text{SO}_2$  monitoring also appeared (396). The response of the detector was reported to be nonlinear to  $\text{SO}_2$  and  $\text{H}_2\text{S}$  because of self-absorption (324).

A dual flame photometric detector allowed simultaneous and selective determination of phosphorus, sulfur, and chlorine containing compounds from a GC column (884). The molecular emission HPO (526 nm) and  $\text{S}_2$  (394 nm) bands were measured in the lower flame, while after reaction of chlorine with In, the InCl emission (360 nm) band was measured in the upper flame. By using a monochromator or selective filters, the indium flame photomet-

ric detector was found to be specific for column effluents containing chlorine, bromine, or iodide (620). The selective detection of sulfur, chlorine, and nitrogen in organic compounds was enhanced by the presence of a copper salt tip fitted to the flame jet (778). All organic compounds containing a heterocyclic atom could be detected under oxidative flame conditions. The response from an alkali flame photometric detector was predominantly negative—*i.e.*, the alkali emission decreased when chromatographic solutes were burned in the flame (45, 619).

The utility of the FPD was seen to be primarily in the identification of crude oils through the use of a dual FID and FPD (6), sulfur containing air-borne pollutants at the ppb level such as  $\text{H}_2\text{S}$  and COS (358), and sulfur containing compounds in coal (194), cigarette smoke (346), and diesel exhaust (315). The selectivity of the FPD was illustrated with a single-step cleanup procedure for pesticides and their metabolites in foods and biological tissues (579) and the detection of Cr in urine at 425.4 nm (729). A lower limit of detection of 0.71 ng of  $\text{B}_{10}\text{H}_{14}$  with a FPD was observed at 546 nm (816). Flame photometric detectors selectively determined organometallics (388) and fluorine down to 43 ng by the formation of CaF (357). Volatile metal fluorides were spectroscopically detected using a low temperature, hydrogen diffusion flame on a capillary burner (191).

The resurgence of interest in microwave discharge detectors was stimulated by the need for more sensitive methods of gas analysis, the need for qualitative identification of chromatographic effluents, and the lack of definitive operational, spectroscopic, and analytical data. The sensitivity of such high frequency discharges for sulfur-containing compounds was  $5 \times 10^{-7}$  vol. % (781). Carbon, nitrogen, and/or sulfur emission in the vacuum ultraviolet from a 2450-MHz microwave source was described by Braun *et al.* (105) at the National Bureau of Standards for the detection of air pollutants. A helium photoionization detector utilizing an electrodeless microwave discharge produced an intense 21-eV ionization source of high stability and reliability. Freeman and Wentworth (277) found the detector sensitivity to be  $1.3 \times 10^{-11}$  gram/ml (Ar) and a linear dynamic range of  $10^4$ .

The major contribution of microwave-excited detectors may be in elemental analysis as they may be operated to give sensitive, simultaneous, selective, and nonselective response (193). A combination elemental selective microwave detector and FID eliminated deposition of carbon within the plasma tube by introducing a permanent bleed of air,  $\text{O}_2$ , or  $\text{N}_2$  (578). Chromatographic identification by the use of inter-element ratios required two atomic lines to determine the relation between the heteroatoms and the number of carbon atoms in a compound (192). The measured ratios were found to be independent of carrier gas flow rate, concentration, and chromatographic peak shape. A 30-MHz plasma was compared to the 2450-MHz source at the same power levels and found to be more sensitive for phosphorus (917). Another radio frequency discharge source (8 MHz) was limited to ppm detection limits because of instability and a high background from  $\text{N}_2$ , NH, NO, and OH band emission (100). A direct current glow discharge detector was developed to detect traces of  $\text{CO}_2$  in purified air (626), and a point discharge detector using Ar as a carrier gas detected less than 0.1 ng of organic solvents (755).

A scanning fluorescence GC detector system which generated both the emission and excitation spectrum, demonstrated sensitivities to anthracene and pyrene of  $1 \times 10^{-11}$  and  $5 \times 10^{-12}$  gram/sec, respectively (275). The basic principles and instrumentation for gas phase fluorescence, and their potential application to the analysis of polynuclear arenes were described by Burchfield, Wheeler, and Bernos (119). They discussed the differences in intensity between the gas and liquid phases and various techniques for increasing the sensitivity.

Absorption spectroscopic methods for organomercury compounds in GC effluents require reduction of all mercury to the elemental state before measurement. Hey (387) reduced organic mercury compounds by passing them through a 20%  $\text{H}_2\text{SO}_4$ -10%  $\text{SnCl}_2$  aqueous solution before introducing the mercury vapor into an atomic absorption spectrophotometer. Longbottom (539) employed

UV absorption at 254 nm after reduction by the CuO nitrogen oxidation method or with a flame.

Schomburg and Henneberg (759) discussed the applications and limitations of MS, NMR, and IR to the identification of chromatographic components. Although an NMR flow cell for GC was developed (862) and Raman spectra were obtained on nanoliter fractions (652), the major emphasis and application of absorption methods was IR.

The advantages of using a cholesteric system for separating sample from carrier gas in a GC/IR system over a gas phase system are that (1) solution spectra are more readily comparable with reference spectra, (2) color shifts occurring on adsorption and desorption onto the liquid crystal film can be monitored to trigger the IR scan, and (3) the delay time errors of gas cells is avoided (522). Fourier transform IR spectra were recorded from flowing and from trapped ( $10^{-2}$ – $10^{-3}$   $\mu$ l) samples (521, 547). Column effluents were also condensed in  $\text{CCl}_4$  and fed into an IR flow cell (409). Because one-minute scans were required, a stop flow technique was used. A new double beam, ratio recording grating IR that scans from 2.5 to 15  $\mu$ m in 6 sec. was designed especially for GC analysis (677). The accuracy of determining components in mixtures by GC/IR was evaluated by Janik (420). Thermal gravimetric analysis and GC/IR were also applied to such complex mixtures as photo-resist remover, ceramic tapes, and protective coatings (188). Other GC/IR applications of current interest included the determination of hydrocarbons in water (428, 464), drugs (519), and anaerobic bacteria species (142).

**Electrochemical.** In his review of specific chromatographic detectors, Littlewood (534) contrasted electrochemical methods to ionization and servomechanisms. Pretorius (692) and Pretorius and Hahn (691) were issued patents for solid electrode detection systems for gas and liquid chromatography. Using a reference electrode, any concentration dependent electrical quantity can be measured such as conductivity, current, capacitance, zeta potential, etc. The use of ion selective electrodes for detecting fluorine, chlorine, and sulfur containing compounds in GC was developed in detail (297).

Selucky (773) discussed the design, specificity, oxidative mode, catalyzed oxidative mode, reduction mode, response, sensitivity, and applications of the electrolytic conductivity detector. A specially treated nickel catalyst was used to implement a conductivity detector with a sensitivity between that of FID and ECD (435). Russian workers measured the conductometric changes on the surface of polar adsorbents of gels during adsorption of gases and referred to this as the sorption conductometric detector (252).

The errors and interferences associated with the electrolytic conductivity determination of chlorine and bromine from pyrolysis of halogenated compounds was found to be due to the formation of  $\text{CN}_2$  (156). The selective response of the detector to chlorinated pesticides in the presence of PCB's was achieved by precise control of the furnace temperature and the  $\text{H}_2$  flow rate (220). The response of a number of sulfur-containing pesticides, some of which also contained chlorine and nitrogen, were investigated using both the pyrolytic and oxidative modes (162). Precautions were given for measuring nitrogen containing pesticides such as s-triazine, substituted urea, carbamate, and miscellaneous herbicides (161). One of the results of two comparative studies of the electrolytic conductivity and alkali flame ionization detectors showed that the former is specific to nitrogen compounds while the AFID responds to various hetero atoms (323, 663).

An improved construction of the flow-through coulometric detector resulted in higher reliability and limits of detection ranging from  $10^{-5}$  to  $10^{-7}$  vol. % for  $\text{H}_2\text{S}$ ,  $\text{SO}_2$ ,  $\text{CS}_2$ ,  $\text{NH}_3$ , etc. (822). High selectivity of the coulometric detector for the above gases was obtained with an oxidative microreactor (506). The major advantages of the detector in air pollution analysis were reported to be linearity and specificity. Its major disadvantages are slow response and relatively low sensitivity (479). DDT and PCB's in soils were found to give a nonlinear response curve (414).

**Computerization.** The rapidly increasing importance and development of laboratory data systems for GC re-

sulted in a large number of symposia, general and descriptive papers, and publication of innovative experiments which would not otherwise be possible. Excellent introductory papers and overviews of computer automation in GC were published by Gill (299, 300), Derge (210), Anderson (21, 22), Ford (265), and Dessey (211). In discussing design philosophies, Deans (198) made the point that a data system should be developed towards the end objective—the utilization of the analytical results. Looking to the future, Anderson (21) pointed toward the increased availability of lower cost hardware and bulk storage, an increasing shift toward firmware and software control, and in-house development of software in the future. The implementation of hierarchical computer systems was also foreseen for collection and processing chromatographic and laboratory data (491). An example of a hierarchical system handling 20 GC's was given (355).

The details of hardware and software considerations have been prerequisite to a thorough understanding of the effects on chromatographic data. Analog vs. digital data transmission was evaluated with respect to the properties of data acquisition components (754). Busch (123) evaluated the relative merits of analog multiplexing to an analog-to-digital converter (ADC) and dedicated ADC's with digital multiplexing. Hardware and software errors, system linearity, and the importance of system design were objectively discussed in a paper by Fozard *et al.* (267). The importance of developing accurate and meaningful algorithms was shown by Chesler and Cram (147, 148) in their treatment of the effect of peak sensing, digitization errors, and random noise on the precision and accuracy of peak shape analysis. Both Gaussian and asymmetrical peaks were considered, and the accuracy of measuring peak areas using different truncation limits, data acquisition rates, and ADC's was given. Reproducibility was shown to be limited by the effects of low frequency noise, regardless of the method of peak area apportionment (266). Although it is possible to obtain results which are not limited by the computer system but by the technology of chromatography, the effects of the sampling interval (485, 487), smoothing parameters, signal-to-noise ratio, deadband limits, peak shape, etc. must be minimized (830).

Annino and Bullock (24) used a pseudo-random sample switching function and successfully developed correlation chromatography. Although their work was directed toward process control, their "proof of principle" will stand as a landmark in the next generation of GC. For highly precise thermodynamic characterization of chemical species and for parameter optimization, an on-line, dedicated GC system dramatically illustrated the concept of "new information" from chromatographic signals (836). The first closed-loop computerization for optimization of experimental conditions was reported which included real-time control of the sampling system, carrier gas flow rate, and temperature (855, 856). This work of Thurman and Burke also represents the enhancement of the power and potential of all chromatographic studies and analyses. Statistical moment analyses of asymmetrical GC peaks can only be measured accurately with digital data systems in order to reflect the time significance of phenomena such as column overloading (169).

Chesler and Cram (149) convoluted Gaussian, exponential, and hyperbolic tangent functions to chromatographic peaks by iterative curve fitting. They pointed out that the method can be extended to the resolution of overlapping peaks and the analysis of complex elution profiles, such as for the measurement of the rate of on-column chemical reactions. The curve fitting program of Clough, Gibb, and Littlewood (160) gave results repeatable to 0.2 to 0.3%. The role of computerization of GC for qualitative analysis by on-line interactive processing of GC-MS data incorporated the experimentalist into the loop to make judgments regarding the selection of data and the mode of data reduction (274). Peak identification by Kovats retention indices and adjusted relative retention times were compared and by means of a satellite IBM 1800 processor into an IBM 360-65, Kovats retention index system was found to be the method of choice (129).

Peak area deconvolution of unresolved peaks remain a difficult problem because of the vast variability of peak

shapes. One algorithm was based on the minimization of a multi-parameter functional form by a combination of descent and linear least squares methods (732). The procedure was highly efficient, even in the case of strongly asymmetrical overlapping peaks. Fitting Gaussian curves (104) is easily done but neither accurate nor valid in practice. Carle (131) derived an equation for calculating the resolution between adjacent peaks for peak heights which vary as much as the dynamic range of the instrument.

Automation of GC should add system reliability as well as ease, speed, and precision of data handling. The former was accomplished by designing a programmable timer which used pre-cut program cards to select the computer program and automation time for each analysis (197). The software capabilities and limitations of data acquisition with digital integrators were compared for time share (415) and on-line mini-computer (83) data reduction. A precision of 1% in the peak area with an analog integrator and digital readout system was developed by eliminating the dead time between measurements (405). Klatt and Carr (488) built a low cost digital integrator, and programmable calculators were shown to be an inexpensive, versatile, and easily programmed data handling system (540).

The rapid development of GC computerization and constantly changing electronic technology has led to a proliferation of descriptions of systems in the literature. The relative merits of off-line systems using time-share computers were described with either fixed program pre-processors (301, 570), ADC's and high speed paper tape punches (380, 556), or hybrid systems with mini-computers (561). System configurations for on-line data handling predominated, perhaps largely because of availability. Large chromatographic laboratories (>8 instruments) were serviced by an IBM 1800 (322) and an IBM 1130 with 8K of core which included feedback control for activating valves, auto-injectors, etc. (180). A 40-channel multipurpose EAI system (693), real-time batch monitoring SIGMA 2 system with core resident peak area allocation programs (513), the Varian Data System acquisition unit which eliminated common-mode voltage effects (905), and the Siemens System 300 which is capable of monitoring 60 GC's with 16K of core (62, 590, 666) were described, and all are indicative of large in-house systems presently in use. The flexibility of the large Mulheim PDP-10 on-line computer system allowed both extended off-line computations and interactive multi-user time sharing (760, 761, 949). A German hierarchical computer system was developed to efficiently use satellite computers for on-line data acquisition in real-time (910). Smaller "mini's" handled GC's which incorporated as many as three detectors and pressure and temperature transducers (140), and were used to automate (391), control (914), and configure total systems (674).

The first commercial application of large scale integrated (LSI) circuits in analytical chemistry evolved the new concept of computing integrators (386). Random access and read-only memories permit the implementation of sophisticated programs such as nonhorizontal base-line corrections, tangential peak splitting, and maintenance of optimum peak and base-line detection sensitivity and noise rejection in a laboratory size package for up to four GC's.

Some of the very interesting analytical applications of GC-computer systems included Burke and Ackerman's (122) surface area measurements by frontal analysis, the determination of the temperature dependence of Kovats retention index (611), the use of simultaneous parallel columns (696), pesticide analysis (863), and Kaiser's (440) ppb trace analysis at the  $\pm 3\%$  (relative standard deviation) error level.

## QUALITATIVE AND QUANTITATIVE ANALYSIS

**Qualitative Analysis—Retention Considerations.** Qualitative analysis in gas chromatography in the classical approach consists largely of comparison of the retention of an unknown component with that of an authentic sample. Another major approach to qualitative analysis involves the combination of GC with other instrumental techniques such as mass spectrometry and infrared spectroscopy. Both approaches will be covered in this review.

The authors have noted a great increase in interest in the use of Kovats retention indices during the past two years for designating the position of an eluted peak in a reproducible manner and for studying the effect of molecular structure on retention. Berezkin (69) reviewed the state of the theory of retention indices and included consideration of Gibbs absorption coefficient. Lorenz and Rogers (542) caution that many factors affect the retention index, and therefore it is not always a dependable means for the characterization of GC behavior. An equation representing the errors from various sources was developed by Takacs *et al.* (841). The dependence of the retention index on temperature is one consideration (218, 611). Thermodynamic proof for Kovats system of retention indices and the relation between retention time and carbon number were outlined (47). The relationship between retention data and molar volume can be used for the identification of organic compounds (929). The need for a polar retention index system based on a polar homologous series was expressed by Gröbler (330), and a series of mono-*n*-alkylbenzoles was used by Louis (544) as standards in a "Quasi-Kovats" system for the reliable identification of aromatic compounds. A retention index system based on homologs of dimethylcyclopolysiloxane was devised (288), and it was found that there is a linear relation between the indices and the number of silicon atoms in the linear and cyclic dimethylsiloxane homologs.

Useful tables of Kovats retention indices were published by several authors. These include a list of 195 commercially available non-olefinic hydrocarbons on Lukopren G 1000, a liquid phase manufactured in Czechoslovakia (543); 109 organosilanes and siloxanes on Apiezon L, silicone oil DC 550, and di-(2-ethylhexyl)sebacate columns (928); 56 substituted chlorosilanes on polymethylsiloxane PMS-20000 and polymethyltrifluoropropylsiloxane FS-303 columns (9); 32 aromatic isothiocyanates on the non-polar and polar columns UCW-98 and DEGS (497); *n*-alkenes, cyclopentenes, and cyclohexenes on squalane, Apiezon L, and polyethyleneglycol 4000 columns (241); permethylated peptides on OV-1 and OV-17 (130); 26 saturated and unsaturated alcohols on 25 liquid phases (911) and 14 diamines on Pennwalt 223 (365).

Interest has been renewed in the use of retention indices in structure determination, the application originally suggested by Kovats for retention indices. Correlations between retention indices and molecular structure were made for benzene and benzene derivatives (168); essential oils (950); mono- and sesquiterpenic hydrocarbons (552); positional isomers of monofunctional, straight-chain C<sub>10</sub> alcohols, acetates, ketones, epoxides, ethers, and olefins (81); branched-chain paraffins (137) and dimethyl paraffins (400); and olefins, cyclic hydrocarbons, and homologs of benzene (844). Retention indices for widely different mono- and poly-functional compounds may be predicted using the assumption that contributions from individual groups are additive (465, 535, 758).

A correlation between chemical structure and retention of C<sub>19</sub> steroids was reported in terms of "steroid numbers" (254). The Rohrschneider concept was recommended by Takacs *et al.* (448, 843), as an extension of retention index measurements since it was shown that the interaction between a solute and the liquid phase can be characterized not only by the difference in retention indices but also by quotients of their indices. The concept of retention boiling point was introduced and suggested as a supplement to Kovats structural identification method (48), and the relationship between boiling points and retention indices of closely similar hydrocarbons was reported in two other publications (807, 809). Retention indices for normal chloro-, bromo-, and iodoalkanes with halogen substitution in the 1-, 2-, and 3-positions were measured on Apiezon L, tricresyl phosphate, and Carbowax 20M (136). The identification of sulfur constituents in crude oil was effected by retention index measurements (7). Stereochemical configurations of various terpenic mono- and di-alcohols were established through retention index measurements (668).

A thorough study of systematic errors in the determination of retention times was made by Goedert and Guiochon (307), and the effect of temperature gradients and fluctuations on gas chromatographic retention data was

reported separately (306). The influence of sample size on the retention of solutes for the case of a variable partition coefficient, or non-linear isotherm has also been tested experimentally (520). Retention times were reported for 45 saturated aliphatic esters on SE-30 (141), for fifty 2- and 4-alkyl and alkenylpyridines on SE-52 (658), and for 23 trimethylsilyl disaccharides on OV-1, OV-17, and OV-25 (378). The separation of nine straight chain undecenes was effected and the components were individually identified on purely chromatographic retention considerations (810).

**Qualitative Analysis—Ancillary Techniques.** Recent developments in GC/IR, GC/MS, and GC/MS/computer were reviewed by Plant (684), and it was noted that such combinations as GC/thermal analysis/IR and GC/MS/IR are also in use. By far the most used ancillary instrument during the past two years has been the mass spectrometer with applications of GC/MS being now published at the rate of approximately 200 papers per year. The increased interest in GC/MS is exemplified by its use within just one governmental agency, the Environmental Protection Agency. It is reported (143) that the success of the first GC/MS installation in 1970 for the identification of pollutants resulted in a proliferation of such systems within the EPA with 19 units operating in August 1972 and an expected increase to 50 units by 1975. The papers reviewed here will be limited largely to those in which fundamental developments in GC/MS and the other ancillary techniques were reported.

Karasek (450) reviewed the interfacing of GC and MS, vacuum and system considerations, computerization, and commercially available units. The complete GC/MS/computer system developed at Cornell employing a Digital Equipment Corp. minicomputer was described by Venkataraghavan and McLafferty (880). A portable, self-sustaining GC/MS system developed by scientists at the Jet Propulsion Laboratories for the 1975 Mars lander with its unique hydrogen carrier gas recycling system was also briefly described (408). More complete details of the hydrogen recycling system are given elsewhere (207). Other GC/MS/computer on-line (109, 384, 395) and off-line (709) systems were reported.

Picogram amounts of drugs may be quantitatively measured with GC/MS and the aid of a computer by scanning the magnetic field repetitively over a narrow mass range of 20–30 amu (49). Computer control of the accelerating voltage allows the determination of isotopic abundance ratios (395). A channel separating device that provides an analog signal for each ion independently was recommended to adapt a low-cost mass spectrometer to a "slave detector" for GC (473). The construction, operation, and practical experience of an accelerating voltage alternator system which provides independent focusing for each mass, magnet stabilization, and a sweep option were described by Klein *et al.* (489), and gave accuracy in carbon isotope ratios of 1 part in 10,000. Comparison was made of selective ion monitoring *vs.* repetitive scanning during GC/MS (592) with the conclusion that selective ion monitoring is capable of greater sensitivity, but the greater versatility of repetitive scanning renders it superior for many applications. A palladium-silver alloy separator and hydrogen carrier gas may be used routinely for analysis of gaseous mixtures in the ppm range (805). The combination of GC and chemical ionization mass spectrometry provides a new mass spectral technique (756). A double-focusing MS9 mass spectrometer was modified for use with GC to permit operation in both chemical ionization and electron impact studies (937).  $^{63}\text{Ni}$  was used as an external MS ionization source allowing the detection of picogram amounts of compounds of biological interest (398). The advantages of a quadrupole MS/GC unit was outlined by Carrico (132), and a quadrupole mass filter was employed by Reeve as a sensitive and selective detector for direct gas chromatographic coupling (708). An improved polyimide ferrule for all-glass GC/MS systems made of Du Pont Vespel SP-1 resin has been used successfully for 12 months, does not cold-flow, and forms helium-tight seals when operating at 50–70 psi pressure and 300 °C temperature (567). A simple device for the introduction of mass calibrant into a GC/MS system was developed (757). The direct coupling of a capillary column

and a mass spectrometer was successful in the analysis of  $\text{C}_{11}$ – $\text{C}_{14}$  reformed petroleum products (835). The means for circumventing the problem that molecular separators operate optimally at only one flow rate, are described in a study of flow programming GC/MS (321). Small glass traps containing 80–100 mg of 100–170 mesh Corning 7930 porous glass have adsorbing efficiency close to 100% for GC fractions (447) and may readily be used for introducing samples to the mass spectrometer. Details are given for the construction and performance of a membrane molecular separator (317), a porous silver membrane separator accommodating a wide range in flow rates (320), and a fritted-glass molecular separator fitted with a remotely operated high-temperature valving system to provide venting of unwanted GC effluent (593).

Fundamental developments of GC/IR include a Ph.D. dissertation by Lephardt (521) in which an IR Fourier transform spectrometer was applied to the analysis of GC effluents. A double-beam, ratio-recording, grating-IR spectrometer that scans from 2.5–15  $\mu\text{m}$  in 6 seconds and can repeat every 8 seconds was designed for "on-the-fly" GC/IR research (677), and a novel "on-the-fly" GC/IR technique proposed by Lephardt and Bulkin (522) traps the solutes on a thin film of a cholesteric liquid crystal mixture coated on the face of an IR cell. Each component is adsorbed as it is eluted from the column, producing a zone that moves across the film, and the spectrum of each effluent is recorded as its zone traverses the film. The combination of preparative GC and micro-infrared spectroscopy was evaluated for the analysis of phenothiazine and related drugs (202). Syn and anti isomers of acetaldoxime were separated by GC, and the IR spectra for the two isomers were determined (431).

Other instrumentation which has been coupled with a gas chromatograph includes centripetal thin-layer chromatography (422) and other forms of TLC/GC coupling (89, 268, 531). A flame photometer for ppm levels of alkyl lead compounds in gasoline (623), a plasma chromatograph (454), NMR with flow cell inlet coupled to a gas chromatograph (862), and a laser-Raman spectrometer with trapping of nanoliter quantities of GC fractions in glass capillary tubes for use in the coaxial (180°) mode of a Cary 81 spectrometer (652).

**Quantitative Analysis.** Fundamental research on new quantitative methods has been rather sparse during the past two years. Derge published a two-part article in which the effect on the reliability of quantitative results of (I) sample preparation, sample introduction, the column characteristics, and detector design and electronics (209) and (II) electronic data acquisition, integrators, and recorders (208) are reviewed. A method was proposed for determination of the full area of chromatographic peaks extending beyond the limits of a recorder chart which is based on a Gaussian curve approximation to the peak shape recorded (733). Barry and Rosie (56, 59) reported that the relative molar response factors for the thermal conductivity detector may be accurately predicted from thermal conductivity coefficients and other physical properties of the chromatographic fraction and the carrier gas. A decrease in the net signal response owing to the background signal in the flame ionization detector from liquid phase bleed was reported by Novak *et al.* (648).

Solid organic compounds, mostly aromatic hydrocarbons, were studied as internal standards for quantitative measurements by determining their retention times on five different columns at temperatures ranging from 125–275 °C (257). The contribution of an overlapping peak to the peak height may be subtracted by a manual method (615). Grushka and Monacelli (342) employed a slope analysis method for recognizing strongly overlapping double chromatographic peaks. McNair and Cooke (582) monitored peak symmetry as a function of increasing sample volume by determination of the statistical moments of skewness and kurtosis. A rapid, easily programmed technique requiring only two data points per peak was developed for resolution of overlapping peaks (718), but the method assumes a Gaussian shaped peak. An algorithm and FORTRAN program for the evaluation of the area of unresolved chromatographic peaks, even those strongly asymmetric, have also been published (732). Other authors have also considered computer de-

convolution of overlapping peaks, resolution of peaks, and peak area measurement (131, 160, 266, 559). The inherent errors involved in computer and integrator methods that simply drop a perpendicular to the base line in allocating areas of overlapping peaks have also been emphasized (486).

## METHODOLOGY

**Carrier Gases.** The influence of carrier gas pressure on column efficiency and stationary phase selectivity has been treated by Russian workers (892). They considered the role of pressure gradients along the column, the effects of turbulent flow, and the measurement of physicochemical properties under supercritical conditions. A mercury displacement pump was used to Jentoft and Gouw (430) to obtain supercritical pressures with CO<sub>2</sub> as the mobile phase in their separation of a mixture of C<sub>8</sub>-C<sub>20</sub> *n*-alkyl bromides. Further experimental work using supercritical mobile phases has been described by Rijnders, Bleumer, and van Krevelen (715).

Unconventional mobile phases such as O<sub>2</sub>, for the determination of small quantities of N<sub>2</sub>, H<sub>2</sub>, and He in reactive propellants (628), and ligand vapors for minimizing dissociation of metal chelates during separation have been reported (283). Mixtures of alcohols, organic acids, and amines were separated on adsorbent columns (GSC) with a minimum amount of tailing by using steam as the carrier gas (642). One group has even used steam with capillary and preparative scale columns to suppress sorption, and thereby elute sharp symmetrical peaks, and to prolong the life of the column packings (734).

Theoretical studies in chromatography have derived equations to characterize the chromatographic processes for separations with a moving temperature field and in the absence of a carrier gas (788, 933). These theories consider the band broadening diffusion and, therefore, the equation for the concentration profile along the column can be derived. Russian workers demonstrated the analysis of low concentration components in mixtures on the basis of chromatography under overloading (793).

**Sampling and Sample Preparation.** The significance and importance placed on sampling in chromatographic analyses in recent years has culminated in the publication of new sampling systems, modifications of others for specific analyses, and the development of devices which are based on chemical and physical phenomena. Thus, it is expected that an increased emphasis on precision, chemical selectivity, sensitivity, fundamental considerations of the sampling process, and applications will be of increasing importance in the future. In the latter case, Harris (368) showed by Raoult's Law that the band width of the sample at the inlet should not contribute significantly to the effluent band width for best resolution of large samples.

The state of the art in precision sampling was reported by Bowen, Cram, Leitner, and Wade (103) in their detailed evaluation of injection peak profiles through a statistical moment analysis. Using computer control of several automated sampling valves, they achieved relative standard deviations of  $\pm 0.1\%$  in the peak area and a reproducibility of 1 part in 120,000 for retention data. Selectivity and sensitivity in sampling has been demonstrated by the use of pre-column concentration and separation techniques. For example, fatty acids in complex biological mixtures were pre-separated by TLC in a closed system where the carrier gas swept the vaporized TLC fractions directly onto a GC column (531). A method for the determination of sub-ppb amounts of lindane by centripetal thin layer chromatography combined with GC was described (422). Polycyclic aromatic hydrocarbons in automobile exhaust were separated into naphthalene through fluoranthene and benanthracene through dibenzopyrene fractions on a Sephadex column prior to GC analysis (326, 327).

A study of the preservation of biological tissue for organochlorine insecticide analysis showed breakdown of *p,p'*-DDT to *p,p'*-TDE occurred in livers stored in formaldehyde, resulting in major error in the analysis (278). Ultrasonic homogenization in the presence of a surfactant

solubilized or stabilized hydrocarbons in water for GC analysis (312). Kaiser (439) enriched volatile compounds from gases by means of a temperature gradient tube. Sorbent materials may be combined with the thermal focusing technique for application to a wider diversity of applications. The method has the additional advantage of being a locally separated collection so as to prevent chemical reaction of the enriched traces with each other.

Precolumns permitting septumless injection minimized the solvent effect by partially venting the solvent during a temperature program of the precolumn (246). A carbon adsorption column efficiently and reproducibly concentrated 11 chlorine and 10 phosphorus pesticides from water (239). However, poor recoveries were reported when the pesticides were stripped with ethyl ether and hexane. Hydrocarbons up to toluene in crude oil were concentrated on a Chromosorb W (20% Apiezon L) precolumn (503). Refrigerated silica gel columns preconcentrated C<sub>2</sub> to C<sub>5</sub> hydrocarbons in air (110), and headspace gases over orange juice were sorbed on Chromosorb 101 because it did not retain water vapor (763). Subtractive GC was useful in determining trace hydrocarbon impurities (636). The sample was injected through a precolumn of 5A Molecular Sieve which adsorbs the principal sample constituent and only the impurities enter the GC column.

High pressure sampling devices were patented (401) and methods were published for vapor-liquid systems up to 1000 psia (477). A sample valve for direct transfer from pressurized sources into the GC (927) and a "syringe gripper" for syringe injection of high pressure gas samples were shown to offer improved performance (704). Other nonconventional sampling systems included a vacuum sample inlet system (381), a method for low pressure sources (538), a vaporizer which was unaffected by pressure changes of the carrier gas or temperature changes of the sample (373), a gas-liquid equilibrium system (562), and sealed source injectors (224).

Wade and Cram (896) illustrated fluidic logic for automated GC sampling. The unique aspect of this injection system was that there were no moving parts. Valves patented by Harris and Harris featured a receiving chamber for high accuracy (366) and a Teflon tube with a reciprocal plunger (367). However, Roper (726) pointed out that the permeability of polytetrafluoroethylene can cause errors in GC analysis by diffusion of materials into the carrier gas and absorption of permanent gases.

Innovations in syringe techniques described sampling moderately pressurized gases and liquids (647), gas syringes heated to 300 °C (478), a syringe dilution method for exponential calibration (263), and an autosampler for solids, liquids, and gases (659). Capsule injection systems for solvent-free operation (5), and other solid sampling systems were developed for biological materials. These included an all-glass system for steroids in body fluids (874), a means of micro-functional group analysis of dried extracts (459), and systems described specifically for steroids (303), steroid hormones (706), and barbiturates in urine (705). Reproducibility of light hydrocarbon analyses with the indium tube technique was found to be superior to syringe sampling (232). The injector of Pease (675) provides for on-column injection of solids without interruption of column gas flow and may be precooled if required. Sealed ampule methods were particularly valuable in avoiding fractionation and loss of volatiles for radiolysis (29) and reaction kinetics studies (392). A new injector design which prevented contamination from the septa proved especially valuable in air pollution, aroma, and trace analysis studies (657). Two methods were described for establishing septum bleeding. In addition to septum conditioning (943), a water cooled septum holder exhibited negligible bleed at temperatures as high as 450 °C (876).

Thermal techniques for sampling ranged from cryogenic concentration and enrichment methods for air samples (703) and head space vapors of food aromas (707, 711) to programmed temperature sample vaporization of low volatility compounds (588). Thermal pyrolysis oxidized sulfur compounds in the ppb range to SO<sub>2</sub> for detection by microcoulometry (235).

**Pyrolysis, Photolysis, and Reaction-GC.** A bibliography of vapor phase thermal fragmentation was prepared

by Sarner (746) and the pyrolysis GC of polymers reviewed (18). Two detailed comparisons were made (15, 903) of the various pyrolyzer types for polymer characterization—vapor-phase, boat, filament, Curie point, ribbon, and laser pyrolyzers—with the conclusion that there is no single design that is optimum for all polymeric samples (903). U.S. (925) and Russian (17) patents have been filed on pyrolysis devices. The Curie-point pyrolysis method is adaptable for the analysis of biological samples such as bacteria, but further development will be necessary for achievement of a useful degree of interlaboratory reproducibility (591). The temperature rise time and a new method of measuring the “true pyrolysis temperature,” a value below the final equilibrium temperature of the heat source and related to the power supplied to the pyrolyzer, was reported by Levy *et al.* (524). Merritt and Robertson (587) proposed that a development of set theory—namely, the Khinchine entropy function—can provide unique diagnostic characteristics when applied to the quantitative distribution of pyrolysis products, but functions calculated in different laboratories vary owing to the interlaboratory nonreproducibility of pyrolysis. Pyrolysis on a tungsten filament using current pulses from a 3000- $\mu$ f condenser provide an impulse energy of 1.35 joules and a pyrolysis time of 0.01–0.1 sec (936). Studies of laser pyrolysis of polymers have continued with the sample applied as a thin film on the surface of a blue cobalt glass rod to facilitate absorption of the laser beam energy (247), and the effects of experimental variables such as laser beam energy and sample film thickness on the distribution of degradation products were determined. In order to absorb light energy, carbon power was mixed with the sodium salts of aliphatic carboxylic acids and subjected to laser thermal degradation (494). Carbon loading, however, affects product distribution of laser-induced degradations (716).

Juvet *et al.* (437) proposed photolytic degradation as a more reproducible substitute for all techniques dependent upon thermal degradation and showed that, in addition to polymer identification, trace additives of antioxidants and plasticizers in polymers may be identified and quantitatively determined. Photochemical degradation was shown to be an ideal approach for the determination of trace pesticide residues on food products (304), where all pesticides investigated—heptachlor, heptachlor epoxide, trans-chlordane, 1-hydroxychlordane, chlordane, nonachlor, dieldrin, aldrin, *p,p'*-DDT, *p,p'*-DDE, and *p,p'*-DDD—gave characteristic degradation patterns. The photolytic degradation of triglyceride esters (735) and of ethyl fluoride at 147 nm (138) were also reported.

Novel applications of pyrolysis GC include differentiation of normal and pathological microbial cells (710), pathogenic microorganisms (359, 585), and other compounds of biological interest such as proteins, carbohydrates, blood components, glandular secretions, muscle, nut oils, and pollen (624); the identification of surfactants by cleavage of hydrophobic groups (526); the determination of combustible carbon in carbonaceous chondrites (248); the identification of flame retardant textile finishes (170) and fiber analysis for forensic purposes (780); other forensic determinations such as the analysis of adhesives, plastics, soils, drugs, fibers (918), and thermosetting acrylic resins used in automotive enamels (558); the pyrolysis of the organometallic compounds, tetramethyltin (433) and trimethylantimony (694); the direct determination of organic carbon in water by reductive pyrolysis (845); the determination of chlorine and bromine (156) and oxygen (676) in organic compounds; the pyrolysis of copolymers with methods developed for distinguishing between copolymers and their homopolymer mixtures (13, 363); and the pyrolysis of sterols (289).

Advances in reaction GC include the combination of GC and reaction microchemistry for the determination of carbon, hydrogen, nitrogen, and oxygen, functional groups, and heteroatoms present (529); the analysis of difficultly saponifiable esters (272) and polymeric amides, urea, and nitriles using alkali-fusion (273), the analysis of sulfur compounds (269, 864), the location of double bonds by means of ozonolysis (407, 434), the quantitative determination of active hydrogen in organic compounds based on reaction with a Grignard reagent (412), the analysis of SO<sub>2</sub> and SO<sub>3</sub> mixtures (219), the analysis of aqueous

phosphate by reaction with bis-(trimethylsilyl)trifluoroacetamide–1% trimethylchlorosilane to form the volatile trimethylsilyl derivative (922); and the determination of alkoxy groups (157) and of primary amino groups (411) in organic compounds.

Precolumns have been used for the selective removal or subtraction of alcohol and carbonyl compounds (184), aldehydes and ketones (362), and nitrogen bases (282). Subtractive methods were used in the determination of several functional groups by reaction of the unknown with chemical reagents within a syringe and analysis of the syringe head space vapors by GC (444).

**Temperature and Flow Programming.** In a review of the basic principles and effects of temperature and flow programming in preparative scale gas chromatography, Kaiser (441) concluded that the combined use of locally limited temperature programming/pressure programming, together with column switching, heart cutting, and back-flushing produced the most effective results.

Experimental studies on the optimization of the program rate in temperature programming (155) and the effect of temperature gradients and fluctuations on retention data were reported (306). A cryogenic trap installed between the flow control system and the injection port was effective in eliminating spurious peaks in sub-ambient programming (594). The use of an equivalent isothermic index,  $I_{T_0/\beta}$ , was proposed for the identification of members of homologous series in linear temperature programming (311). Series of alkanals, 2-alkenals, 2-alkanones, and 2,4-alkadienals were identified on this basis in fish.

A linear relationship between relative retention program temperature data and the logarithm of isothermal relative retention data for adsorption gas chromatography was derived by Takemura (846) in a continuation of his earlier studies. Franck *et al.* (270) showed that evaporation enthalpies could be determined by linear temperature programming with an error of less than 2% when the carrier gas flow rate is held constant.

Longitudinal temperature gradients in programmed temperature gas chromatography were studied theoretically and experimentally. Effects of the rate of change of the gradient, program rate, column length, and initial gradient temperature on retention times were investigated and found to agree with theoretical predictions (173, 253).

The dependence of retention indices, peak heights, and peak areas on the carrier gas flow rate showed that qualitative and quantitative analysis can be carried out under conditions of flow programming gas chromatography (52). Retention indices were found to be independent of carrier gas flow rates up to 500 ml/min. but because peak heights and areas are influenced by a change in the carrier gas flow rate, an internal standard was required for quantitation. Grayson, Levy, and Wolf (321) cited the use of flow programming in GC-MS where column bleed is normally a deleterious effect. In order to circumvent the problem of a change in molecular separator efficiency with flow rate, they suggest various splitter arrangements and means of providing an auxiliary carrier gas supply.

**Trapping.** Condensing glass capillary tubes (111) and electrostatic precipitating devices (90) have been granted patents as fraction collection devices for gas chromatography. A vapor phase collection system reported by Verdin (881) prevents contamination by solvents or adsorbents by trapping the effluent in a 50-ml glass coil at room temperature.

On-line coupling of gas chromatographs and mass spectrometers has largely eliminated the need for fraction collection and trapping. The necessity for identification in trace organic analysis obviates off-line methods which are not effective for quantities less than 10  $\mu$ g, such as cryogenic trapping (804) and adsorption on porous glass traps (447, 455). The latter method has the additional disadvantage of increasing the back pressure on the column and causes an imbalance in non-destructive detectors such as thermal conductivity.

## MISCELLANEOUS

**Physical-Analytical Measurements.** A number of studies have been completed related to the measurement of the heats of adsorption using gas chromatographic

methods. A new method was described and evaluated for the simultaneous determination of the adsorption isotherms of two components in gas-solid systems (295), and a Russian patent was issued for a chromatographic method for the estimation of adsorption isotherms of gases and vapors (860). Okamura and Sawyer (653, 654) studied the relationships between molar refraction, dipole moment, and specific interactions, and differential enthalpies, entropies, and free energies of adsorption on sodium sulfate modified alumina and silica columns. A non-static impulse method was used for determination of adsorption and desorption rate constants (931). Plots of the logarithm of the retention of hydrocarbons and fluorocarbons on paraffin and ester liquid phases *vs.* the reciprocal of the absolute column temperature near the melting point of the liquid phase suggests an apparent increase in the melting point of the liquid phase film in direct contact with the support (775). A method was presented for evaluation of the contributions to solute retention of bulk liquid solution, solid adsorption, and liquid-gas interface adsorption (827). Two methods for obtaining meaningful partition coefficients (or retention volumes) from chromatographic peaks which result from concurrent solution and adsorption partitioning mechanisms were compared by Liao and Martire (527) and an alternative approach was suggested. Adsorption isotherms on a liquid phase-solid adsorbent surface have been measured (71). Measurements were also made of heats of adsorption for C<sub>4</sub>, C<sub>5</sub>, and C<sub>6</sub> hydrocarbons on cobalt oxide (617), heats of adsorption and Henry constants for C<sub>1</sub>-C<sub>6</sub> alkanes, alkenes, alkadienes, alkynes, cyclanes, and cyclenes (443), differential heats and entropy of adsorption and the change in the chemical potential of C<sub>6</sub>-C<sub>10</sub> alkenes (240) and the isotherms and isosteric heats of adsorption in the region of very low surface coverages for propylamine and acetic acid (214) on graphitized thermal carbon black. Henry's law constants, isosteric enthalpies, and potential energies of adsorption, and surface area of activated coconut shell carbon using nitrogen and methane (102) and the adsorption heats of carbon monoxide and benzene on various Zeolites (96) were also measured.

Several workers have made use of frontal analysis chromatography in physical-analytical measurements since this procedure is among the best chromatographic approaches for studying solution effects in finite concentration ranges (145). Surface areas and adsorption isotherms were measured by computer analysis of frontal analysis data by Burke and Ackerman (122). Liquid surface adsorption effects for the system 2-propanol in *n*-heptadecane (667) and adsorption isotherms and heats of adsorption of water and methanol on Porapak R (673) were among the investigations made using the frontal chromatography approach.

Korol (499) prepared a comprehensive review in Russian citing 152 references to the determination of thermodynamic functions by GC and including such topics as the evaluation of molar entropies, heats, free energies, and activity coefficients, the frontal analysis method, and the thermodynamics of solution in medium and high concentrations. The reliability of activity coefficients and partial molar excess enthalpies (920) and of heats of vaporization (221) measured by GC was evaluated using statistical methods. The variation of the free enthalpy of vaporization of the solute with the pressure of the carrier gas along the column was considered (1). Equations were derived in which partition coefficients, partial molar enthalpies, entropies, and free energies of solution, relative activity coefficients, and other thermodynamic quantities were estimated using Kovat's retention index measurements (309). The accuracy of such determinations was considered in another publication (310) and compared with the evaluation of these same functions using specific retention volumes. A new procedure was developed for the estimation of the surface area of adsorbents from the second and third gas-solid Virial coefficients measured gas chromatographically (737). The dependence of activity coefficients on concentration may be predicted using a simplified version of Wilson's equation for excess free energy (404). Roberts and Hawkes (717) claim current definitions of activity coefficient based on mole fraction are unsatisfactory for polymeric liquid phases, and they propose a definition independent of molecular weight of the liquid phase.

Functional group contributions to liquid phase activity coefficients for binary systems have been established from retention measurements (752). The GC method for evaluating activity coefficients was simplified so that it may be used as an experiment for undergraduate physical chemistry students (55). Sorption-desorption thermodynamics was evaluated for 13 low molecular weight compounds on two polymers used in packaging, polyethylene and polyvinyl chloride (877).  $\Delta S^\circ$ ,  $\Delta F^\circ$ , and  $\Delta H^\circ$  of solution for C<sub>5</sub>-C<sub>9</sub> hydrocarbons in dinonylphthalate (890), the saturation vapor pressure of perfumes (895), and sorption thermodynamics for several solutes on various mixtures of the binary liquid phase system, 2,4,6-trinitrotoluene + 1,2,3-tris- $\beta$ -cyanoethoxypropane (891) were measured by Russian workers.

Purnell (697) has been granted a U.S. patent for the use of a mixture of two selected liquid phases at such a composition that the apparent partition coefficient of a solute on the mixture is constant over an extended range in temperature. The aqueous solubilities of *n*-alkyl-*p*-aminobenzoates were determined by GC in the evaluation of partition coefficients of these materials in the silicone oil-water and the hexane-water solvent systems (930). The evaluation of equilibrium constants for complex-forming reactions between a volatile solute and a non-volatile reagent dissolved in a stationary liquid phase was considered in some detail by Eon, Pommier, and Guiochon (244), and the association constants of complexes formed between silver salts in ethylene glycol and substituted alkenes were measured (204). Vapor-liquid equilibrium constants for methane and carbon dioxide in the CH<sub>4</sub>-CO<sub>2</sub>-*n*-octane system were measured at several subambient temperatures (477). Heats and entropies of isomerization and isomerization equilibria of positional and stereoisomers of hexene were evaluated (702). Diffusion of solutes in long-chain solvents was the subject of another investigation (124) in which equations were developed relating carbon number and temperature to diffusivities of *n*-alkanes in methyl silicones and *n*-alkanols in polyethylene glycols. The effect of salts on the diffusion coefficients of non-electrolytes in aqueous solutions (852) and the effect of lithium chloride on the solubility,  $\Delta G^\circ$ ,  $\Delta H^\circ$ , and  $\Delta S^\circ$  of alcohols, esters, and ketones in polyethylene glycol 400 (79) was studied.

Other physical-analytical studies include the thermodynamic stability of monoethylalkanes (618) and isomerization equilibria for methylpentanes and methylheptanes (721) over aluminum halides, the kinetics of the homogeneous gas phase elimination of ethylene from triethylaluminum (163), the kinetics and mechanism of the reaction of fluorine with trichlorofluoromethane (264), the determination of the relative esterification rates of the isomeric hexanoic acids (803), and the kinetics for the decomposition of tetraalkylammonium salts in chloroform and acetone (493).

**Inorganic GC.** The analysis of inorganic and organometallic compounds by GC continues to be a very active area of investigation. Anvayer and Drugov (25) reviewed the literature on the analysis of inorganic substances by gas chromatography and covered the metals and their oxides, hydrides, halides, chelates, solid non-metals, and isotopes. The direct determination of metals in alloys is not common; however, a super high-temperature gas chromatograph, which has operated successfully at temperatures up to 1350 °C, was applied to the quantitative determination of magnesium and zinc in aluminum alloys (811). Anion analysis (carbonate, sulfite, and sulfide) was also reported (576).

Gas chromatographic separations of inorganic compounds are generally performed with the metals as halides, hydrides, chelates, or organometallics. An electron capture detector (107) and a thermal conductivity detector (108) were tried for inorganic chlorides and organometallic chlorides. GC has been used in the quantitative on-line analysis of chlorine, boron trichloride, and boron nitride (217). Two Russian groups (510, 518) independently studied the determination of trace organic impurities in AsCl<sub>3</sub>. The nonideality of metal chloride vapors with a comparison of theoretical and experimentally measured second virial coefficients was examined by Zado and Juvet (941) in a study of the separation of SbCl<sub>3</sub> and MoCl<sub>5</sub> by GC. Chesler and Juvet (150) showed that anion exchange



of halides may be performed rapidly and quantitatively on a chromatographic column, and these authors investigated the advantages of metal iodide and metal bromide separations. Deans and coworkers (191) reconfirmed the separation of a number of metal fluorides using Kel-F oil as liquid phase and a flame photometric detector. The separation efficiency, retention time, elution order, and other features for uranium hexafluoride and other corrosive compounds were shown (36) to be affected by the type of polymer support used—Chromosorb T, Fluoropak 80, Soreflon 5A, and Voltaef 300LD. Sulfur hexafluoride is eluted before oxygen using a 5A Molecular Sieve column at 23 °C and was recommended by Simmonds, Lovelock, and coworkers (794) as a meteorological tracer. Xenon difluoride and xenon tetrafluoride were separated on Kel-F 10 oil using PTFE or MgF<sub>2</sub> as the solid support (11). Sokolov and Vakin (813) designed a flame ionization detector with quartz jet for use with metal chlorides and certain metals at temperatures up to 860 °C. The value of the flame photometric detector for metal containing compounds was reconfirmed by Aue and Hill (40–42, 388), and operation with a hydrogen-rich flame in conjunction with a regular flame ionization detector was evaluated. Attempts to measure the ratios of chlorine, iodine, bromine, sulfur, and phosphorus emission to carbon emission and the factors affecting these ratios with the microwave-excited emission detector was described by Dagnall *et al.* (192). Mixtures of SiCl<sub>4</sub>, Cl<sub>2</sub>, CoCl<sub>2</sub>, Ar, N<sub>2</sub>, CO, and CO<sub>2</sub> were analyzed in a 7-minute period using three columns and three separate detectors (61).

Several metal hydrides may also be eluted chromatographically. Two independent Russian groups have published on the determination of trace organic and inorganic impurities in arsine (612, 954). Decaborane, a substituted boron hydride, was eluted from an OV-1 column and detected in amounts of 0.71 ng using a flame photometric detector at 546 nm (816). These same authors have separated B<sub>2</sub>H<sub>6</sub>, B<sub>5</sub>H<sub>9</sub>, B<sub>6</sub>H<sub>10</sub>, and B<sub>10</sub>H<sub>14</sub> on OV-17 (815). Residues of phosphine present in sugar cane grown in soils treated with zinc phosphide rodenticide may be detected to the 20-pg level with a flame photometric detector (719).

A number of papers were published this biennium on the separation of volatile metal chelates. The ternary iron(II) and iron(III) adducts of 1,1,1,5,5,5-hexafluoro-2,4-pentanedione and tri-*n*-butyl phosphate can be eluted (858). Thenoyltrifluoroacetates of beryllium(II), aluminum(III), scandium(III), vanadium(IV), chromium(III), cobalt(II), nickel(II), and copper(II) may be quantitatively eluted from 1% loaded columns of DC 710 (416). Other studies include the elution of  $\beta$ -diketone, monothio- $\beta$ -diketone, bidentate  $\beta$ -ketoamine and tetradentate  $\beta$ -ketoamine complexes of copper, nickel, palladium, and platinum (66); the determination of trace amounts of nickel, 5  $\times$  10<sup>-11</sup> gram, as the monothiotrifluoroacetylacetonate (54), and the determination of chromium with a flame photometric detector at 425.4 nm (729). The asymmetry of chromatographic peaks, often observed with metal chelates and attributed to dissociation of the chelates in the liquid phase, may be eliminated by use of a carrier gas containing the ligand vapor (283). The separation of rare earth elements continues to receive interest with work reported on the thermogravimetry and GC of rare earth  $\beta$ -diketones (871), rare earth metal pivaloyltrifluoroacetone adducts of tributyl phosphate or trioctylphosphine oxide (872), the separation of the lanthanides, UO<sub>2</sub><sup>2+</sup>, and Th(IV) complexed with mixed ligands, dodecafluoro-4,6-octanedione or decafluoro-3,5-heptanedione in combination with organic neutral donors such as tri-*n*-butylphosphate, tri-*n*-butylphosphine oxide, or di-*n*-butylsulfoxide (791, 792), and other lanthanide mixed-ligand complexes (120, 121). Meloan and coworkers (285, 641) report a direct and a differential method for determining the water associated with metal chelates and solvation numbers in various solvents. Ultramicro amounts of selenium in "pure" tellurium were determined by GC analysis of the 4-nitro-*o*-phenylenediamine complex (784) and picogram amounts of chromium in blood plasma and serum and of beryllium in an environmental residue by GC/MS of a trifluoroacetyl derivative (926).

The determination of methylmercury in fish by GC was the subject of several investigations. The procedure of Westöo was tested over a 1-year period and showed a limit

of detection for methylmercury of 0.02 ppm using an electron capture detector and certain modifications of the extraction and cleanup steps (445). Another procedure is said to be sensitive to 0.01 ppm (870). The procedure used for methylmercury at the Pitlochry freshwater fisheries in Scotland was also described (394). Bacterial degradation of methylmercury in lake sediments was measured by analyzing headspace for methane, a degradation product (817). Inorganic mercury may be determined by GC by conversion to a phenylmercury salt (436). When phenylmercury salts are injected into a gas chromatograph, they are converted in the injection block into diphenylmercury as the chief product (63, 228). A novel combustion-detection technique which avoids the conversion of dialkylmercurials to their salts for electron capture detection was developed for the analysis of raw river water (229) and applied for the determination of four homologous dialkylmercury compounds. A mercury-specific detector making use of the absorbance of mercury resonance radiation at 253.7 nm after conversion to the elemental state was applied to GC (539). Exchange reactions were studied gas chromatographically of bis-triethylgermanyl/mercury with organotin halides (507). Organoarsenic compounds (175, 176, 769) and organosilicon compounds (8, 139, 797, 798, 831, 833) were eluted gas chromatographically, the vast majority of this work with organosilicone compounds being conducted by Russian scientists in the past two years. Automated purity control of chlorosilanes using an on-line Russian process control gas chromatograph was disclosed (46) and the gas-liquid critical temperatures of polydimethylsiloxanes with two to eight silicon atoms per molecule was reported (938). Umilin and coworkers (867) measured the temperature dependence of vapor pressure of bis-arenyl- $\pi$ -complexes of vanadium and successfully eluted various ethylbenzoyl-molybdenum organometallics gas chromatographically (866). Tin (584), selenium and tellurium (262), chromium (467), and thallium (695) organometallic compounds are among those which have now been evaluated gas chromatographically, and research on the elution of nitrogen oxides (76, 334) and organophosphates, and other phosphorus esters was active (399, 771).

Finally, a few interesting and novel applications include the GC determination of penicillins (389), the identification and quantitative determination of microgram amounts of "street heroin" (212, 625), hallucinogens (779), and the drugs in opium extract (64), a GC/MS reference data system with MS base peak index for 133 drugs of abuse (259), and a rapid ( $\leq$  3 hr) analytical method for emergency drug analyses (796). The analysis of the C<sub>15</sub>-C<sub>36</sub> *n*-alkanes in air particulate matter (377), the identification of spilled residual fuel oils (464), and the characterization of organic components in municipal water supplies (490) are representative of the many applications of GC to pollution studies. The artificial sweetening agents, saccharine, cyclamate, and dulcin, may be simultaneously analyzed (498). Numerous steroids in umbilical cord blood plasma (511) and in Florida Spanish Moss (35) were separated by GC. Chromatographic methods have been developed (728) for the analysis of protein and peptide structures making use of the 2,4-dinitrofluorobenzene derivatives of N-terminal amino acids, and North Atlantic Ocean Waters provided a source for the analysis of nanomolar quantities of free amino acids (685). The volatile components in roast beef, roast beef drippings (528), and 17 varieties of black pepper (713) were the subjects of other studies. Trimethylarsine (176), dimethylselenide and dimethyltelluride (262) are formed from the microorganism, *Candida humicola*, and a strain of *Penicillium*, respectively, found in raw sewage. The components of riot control agents may be separated and determined using GC methods (944). Gas chromatographic studies of the anal gland secretion of the red fox by Fox (10), the identification of the components of the sex odor in pork (853) and the two synergistic sex pheromones of the summer fruit tortrix moth, *adoxyphes orana* (583), as well as the analysis of fluphenazine and its sulfoxide in the urine of chronic schizophrenic patients (474) and the identification of the malodorous component of a schizophrenic's sweat (801) are ample examples of the great versatility and importance of this analytical tool in many fields of endeavor.

## LITERATURE CITED

- (1) Abdeddam, K., Tabeb-Ben-Diab, S. A., Vergnaud, J. M., *C. R. Acad. Sci.*, **272**, 1408 (1971).
- (2) Ackman, R. G., *J. Chromatogr. Sci.*, **10**, 506 (1972).
- (3) *Ibid.*, p 560.
- (4) Acuna, A. U., Garcia Dominguez, J. A., Molera, M. J., Santiuste, J. M., *An. Real Soc. Espan. Fis. Quim., Ser. A*, **67**, 101 (1971).
- (5) Adams, R. F., Purcell, J. E., *Chromatogr. Newslett.*, **1**(1), 1 (1972).
- (6) Adlard, E., Matthews, P., *Nature (London)*, **233**(9), 83 (1971).
- (7) Agrawal, B., Tesarik, K., Janak, J., *J. Chromatogr.*, **65**, 207 (1972).
- (8) Ainshtein, A. A., Pavlikova, G. P., Syavtsillo, S. V., Tseitlin, Y. P., *Khim. Prom.*, **48**, 395 (1972).
- (9) Ainshtein, A. A., Shulyatieva, T. I., *Zh. Anal. Khim.*, **27**, 816 (1972).
- (10) Albone, E., Fox, M., *Nature, (London)*, **233**, 569 (1971).
- (11) Aleinikov, N. N., Sokolov, D. N., Korsunskii, B. L., Dubovitskii, F. I., *Dokl. Akad. Nauk SSSR*, **196**, 1341 (1971).
- (12) Alekseeva, A., Aerov, M., *J. Chromatogr.*, **77**, 131 (1973).
- (13) Alekseeva, K. V., *Zh. Anal. Khim.*, **27**, 386 (1972).
- (14) Alekseeva, K. V., Strel'nikova, I. A., Yakovleva, T. S., *ibid.*, **25**, 987 (1970).
- (15) Alexeeva, K., Khramova, L., *J. Chromatogr.*, **69**, 65 (1972).
- (16) Aliev, M. I., Fisher, S. I., Shirinbekova, F. I., Imanov, Z. T., USSR Patent **309,294** (Sept. 20, 1971).
- (17) Alishoyev, V. R., Berezkin, V. G., Blinov, A. S., Kalmanovskii, V. I., Komarova, Y. P., Malyshev, A. I., Rostovtseva, Y. Y., Savinov, I. M., Chernov, A. V., Eventova, I. L., Yashin, Y. I., Mirzabayev, G. A., USSR Patent **284,418** (Jan. 27, 1971).
- (18) Alishoyev, V. R., Berezkin, V. G., Malyshev, A. I., *Zh. Anal. Khim.*, **27**, 1035 (1972).
- (19) Anderson, D. G., Ansel, R. E., *J. Chromatogr. Sci.*, **11**, 192 (1973).
- (20) Anderson, D. M. W., Dea, I. C. M., Hendrie, A., *Talanta*, **18**, 365 (1971).
- (21) Anderson, R. E., *Chromatographia*, **5**(2/3), 105 (1972).
- (22) Anderson, R. E., *J. Chromatogr. Sci.*, **10**, 8 (1972).
- (23) Andrews, M., Schroeder, D., Schroeder, J., *J. Chromatogr.*, **71**, 233 (1972).
- (24) Annino, R., Bullock, L. E., *Anal. Chem.*, **45**, 1221 (1973).
- (25) Anvayer, B. I., Drugov, Y. S., *Zh. Anal. Khim.*, **26**, 1180 (1971).
- (26) Araki, S., Suzuki, S., Iwahara, H., Takahata, Y., *Kogyo Kagaku Zasshi*, **74**, 647 (1971).
- (27) *Ibid.*, p 651.
- (28) Araki, S., Suzuki, S., Watabe, K., Takahata, Y., *Bunsei Kagaku*, **20**, 1200 (1971).
- (29) Armenante, M., Santoro, V., Spadaccini, G., *J. Chromatogr. Sci.*, **9**, 510 (1971).
- (30) Arsenault, G. P., Doihun, J. J., Biemann, K., *Anal. Chem.*, **43**, 1720 (1971).
- (31) Arutyunov, Ye. I., Zibrova, L. P., Karpov, Ye. F., Karbanov, Ye. M., Kravchenko, V. S., Korlyakov, G. A., *Zavod. Lab.*, **38**, 160 (1972).
- (32) Ashes, J. R., Haken, J. K., *Anal. Chem.*, **45**, 1131 (1973).
- (33) Askew, W. C., *ibid.*, **44**, 633 (1972).
- (34) Askew, W. C., Maduskar, K. D., *J. Chromatogr. Sci.*, **9**, 702 (1971).
- (35) Atollah, A., Nicholas, H., *Phytochemistry*, **10**, 3139 (1971).
- (36) Aubeau, R., Blandenet, G., Leccia, F., *Chromatographia*, **5**, 240 (1972).
- (37) Aue, W., Gerhardt, K., Lakota, S., *J. Chromatogr.*, **63**, 237 (1971).
- (38) Aue, W. A., Hastings, C. H., Kapila, S., *Anal. Chem.*, **45**, 725 (1973).
- (39) Aue, W. A., Hastings, C., Kapila, S., *J. Chromatogr.*, **77**, 299 (1973).
- (40) Aue, W. A., Hill, H. H., Jr., *Anal. Chem.*, **45**, 729 (1973).
- (41) Aue, W. A., Hill, H. H., Jr., *J. Chromatogr.*, **70**, 158 (1972).
- (42) *Ibid.*, **74**, 319 (1972).
- (43) Aue, W. A., Kapila, S., Gerhardt, K., *ibid.*, **78**, 228 (1973).
- (44) Aue, W. A., Kapila, S., *J. Chromatogr. Sci.*, **11**, 255 (1973).
- (45) Aue, W. A., Moseman, R., *J. Chromatogr.*, **61**, 35 (1971).
- (46) Avdonin, G. V., Alekseyeva, Z. I., Frangulyan, L. A., Palamarchuk, N. A., Svyatsillo, S. V., Merkulov, V. D., *Khim. Prom.*, **48**, 356 (1972).
- (47) Bach, R. W., Doetsch, E., Friedrichs, H. A., Marx, L., *Chromatographia*, **4**, 459 (1971).
- (48) *Ibid.*, p 561.
- (49) Baczynskij, L., Duchamp, D. J., Zieserl, J. F., Axen, U., *Anal. Chem.*, **45**, 479 (1973).
- (50) Baerwald, G., Prucha, J., *Brauwissenschaft*, **24**, 397 (1971).
- (51) Bagirov, R. A., Kuliev, A. M., Farkhadov, T. S., *Khim. Tekhnol. Topl. Masel.*, **17**(7), 51 (1972).
- (52) Balla, J., Takacs, J., Mazor, L., Kaplar, I., "Proc. Third Anal. Chem. Conf.," Budapest, Vol. 1, 1971, pp 285-89.
- (53) Bardyshev, I. I., Komshilov, Yu. N., Pertsovskii, A. L., *Zavod. Lab.*, **37**, 1438 (1971).
- (54) Barratt, R. S., *Proc. Soc. Anal. Chem.*, **9**(4), 86 (1972).
- (55) Barrett, R., Stewart, T., *J. Chem. Educ.*, **49**, 492 (1972).
- (56) Barry E., *Diss. Abstr.*, **32**, 1407B (1971).
- (57) Barry, E. F., Fischer, R. S., Rosie, D. M., *Anal. Chem.*, **44**, 1559 (1972).
- (58) Barry, E. F., Rosie, D. M., *J. Chromatogr.*, **59**, 269 (1971).
- (59) *Ibid.*, **63**, 203 (1971).
- (60) Barry, E., Trakimas, R., *ibid.*, **73**, 226 (1972).
- (61) Basu, P. K., King, C. J., Lynn, S., *J. Chromatogr. Sci.*, **10**, 479 (1972).
- (62) Baudisch, J., *Chromatographia*, **5**(2/3), 79 (1972).
- (63) Baughman, G., Carter, M., Wolf, N., Zepp, R., *J. Chromatogr.*, **76**, 471 (1973).
- (64) Bechtel, A., *Chromatographia*, **5**, 404 (1972).
- (65) Belcher, R., Majer, J. R., Stephen, W. I., Thomson, I. J., Uden, P. C., *Anal. Chim. Acta*, **50**, 423 (1970).
- (66) Belcher, R., Martin, R. J., Stephen, W. I., Henderson, D. E., Kamalizad, A., Uden, P. C., *Anal. Chem.*, **45**, 1197 (1973).
- (67) Belgian Society of Pharmaceutical Science, "Sixth International Symposium on Chromatography and Electrophoresis," Ann Arbor Sci. Publishers, Ann Arbor, Mich., 1971.
- (68) Belyakova, L. D., Kiselev, A. V., Soloyan, G. A., *Zh. Anal. Khim.*, **27**, 1182 (1972).
- (69) Berezkin, V. G., *Usp. Khromatogr.*, **1972**, 215-25.
- (70) Berezkin, V. G., Alishoyev, V. R., Nemirovskaya, I. B., "Gas Chromatography in Polymer Chemistry," Nauka, Podmosnenski per. 21, Moscow, 1972.
- (71) Berezkin, V. G., Gavichev, V. S., Zorina, G. P., *Izv. Akad. Nauk SSSR, Ser. Khim.*, **1972**, 1975.
- (72) Berezkin, V. G., Korolev, A. A., Smirnov, A. G., *Zavod. Lab.*, **38**, 518 (1972).
- (73) Berezkin, V. G., Nikitina, N. S., *Usp. Khim.*, **40**, 927 (1971).
- (74) Berezkin, V. G., Shlyakhov, A. F., Bolotov, G. M., *Zavod. Lab.*, **38**, 518 (1972).
- (75) Berezkin, V. G., Tatarinskii, V. S., translated from Russian by Bradley, J. E. S., "Gas Chromatographic Analysis of Trace Impurities," Plenum, New York, 1973.
- (76) Beskova, G. S., Filipov, V. S., *Zavod. Lab.*, **38**, 154 (1972).
- (77) Bethea, R. M., Bentsen, P. C., *J. Chromatogr. Sci.*, **10**, 575 (1972).
- (78) Bethea, R. M., Bray, J. M., *ibid.*, **9**, 758 (1971).
- (79) Betti, A., Dondi, F., Lodi, G., Bighi, C., *J. Chromatogr.*, **68**, 59 (1972).
- (80) Bhattacharjee, A., Basu, A., *ibid.*, **71**, 534 (1972).
- (81) Bierl, B. A., Beroza, M., Aldridge, M. H., *J. Chromatogr. Sci.*, **10**, 712 (1972).
- (82) Binder, H., Linder, W., *J. Chromatogr.*, **77**, 175 (1973).
- (83) Bischet, G., Knechtel, G., *Chromatographia*, **5**, 166 (1972).
- (84) Blades, A. T., *J. Chromatogr. Sci.*, **10**, 693 (1972).
- (85) *Ibid.*, **11**, 251 (1973).
- (86) *Ibid.*, p 267.
- (87) Blagoy, Yu. P., Zhun', G. G., Zimoglyad, B. N., Chuprina, G. O., *Dokl. Akad. Nauk SSSR*, **201**, 1134 (1971).
- (88) Blomstrand, R., Gurtler, J., Liljequist, L., Nyborg, G., *Anal. Chem.*, **44**, 277 (1972).
- (89) Bloom, P., *J. Chromatogr.*, **72**, 35 (1972).
- (90) Bloomer, J. L., U.S. Patent **3,608,276** (Sept. 28, 1971).
- (91) Blu, G., Jacob, L., Guiochon, G., *J. Chromatogr.*, **61**, 207 (1971).
- (92) Blu, G., Lazarre, F., Guiochon, G., *Anal. Chem.*, **45**, 1375 (1973).
- (93) Blumer, M., *ibid.*, p 980.
- (94) Bocek, P., Janak, J., *Chromatogr. Rev.*, **15** (2/3), 111 (1971).
- (95) Bogdanchenkov, A. I., Vigdergauz, M. S., *Khim. Tekhnol. Topl. Masel.*, **16**(8), 53 (1971).
- (96) Bogomolov, V. I., Mirzabekova, N. V., Isakov, Ya. I., Golender, L. O., Minachev, Kh. M., *Izv. Akad. Nauk SSSR, Ser. Khim.*, **1972**, 1697.
- (97) Bogoslovskii, Yu. N., Razin, V. L., *Zh. Fiz. Khim.*, **45**, 2490 (1971).
- (98) Bollman, D. H., Mortimore, D. M., *J. Chromatogr. Sci.*, **10**, 523 (1972).
- (99) Boogaerts, T., Verstappe, M., Verzele, M., *ibid.*, p 217.
- (100) Boos, L. E., Winfordner, J. D., *Anal. Chem.*, **44**, 1020 (1972).
- (101) Bothe, H. K., "Aspects in Gas Chromatography," H. G. Struppe, Ed., Akad. Verlag, Berlin, 1971, pp 6-16.
- (102) Boucher, E., Everett, D., *Trans. Faraday Soc.*, **67**, 2720 (1971).
- (103) Bowen, B. E., Cram, S. P., Leitner, J. E., Wade, R. L., *Anal. Chem.*, **45**, 2185 (1973).
- (104) Braswell, E., *Anal. Biochem.*, **44**, 58 (1971).
- (105) Braun, W., Peterson, N. C., Bass, A. M., Kurylo, M. J., *J. Chromatogr.*, **55**, 237 (1971).
- (106) Brazhnikov, V. V., Mukhin, L. M., *Chromatogr. Rev.*, **15**, 151 (1971).
- (107) Brazhnikov, V. V., Sakodinsky, K., *J. Chromatogr.*, **66**, 361 (1972).
- (108) Brazhnikov, V. V., Sakodinsky, K., *Zh. Prikl. Khim.*, **43**, 2247 (1970).
- (109) Briggs, P., Dix, D., Glover, D., Kleinmann, R., *Amer. Lab.*, **4**(9), 57 (1972).
- (110) Brocco, D., Cantuti, V., Moscatelli, F., *Riv. Combust.*, **25**(4), 164 (1971).
- (111) Brownlee, R. G., U.S. Patent **3,623,843** (Nov. 30, 1971).
- (112) Bruner, F., Ciccio, P., DiCorcia, A., *Anal. Chem.*, **44**, 894 (1972).
- (113) Bruner, F., Ciccio, P., Rastelli, R., *J. Chromatogr.*, **77**, 125 (1973).
- (114) Bruner, F., DiCorcia, A., Goretti, G., Zelli, S., *ibid.*, **76**, 1 (1973).
- (115) Bruner, F., Liberti, A., Possanzini, M., Alegrini, I., *Anal. Chem.*, **44**, 2070 (1972).
- (116) Buechi, J., Lorini, V., Perlia, X., *Pharm. Acta Helv.*, **47**, 15 (1972).
- (117) Bulow, M., Grossman, A., Schirmer, W., *Chem. Tech. (Berlin)*, **23**, 33 (1971).
- (118) Bunting, T., *Diss. Abstr.*, **31**(11), 6463B (1971).
- (119) Burchfield, H. P., Wheeler, R. J., Bernos, J. B., *Anal. Chem.*, **43**, 1976 (1971).
- (120) Burgett, C. A., Fritz, J. S., *ibid.*, **44**, 1738 (1972).
- (121) Burgett, C., Fritz, J., *J. Chromatogr.*, **77**, 265 (1973).
- (122) Burke, M. F., Ackerman, D. G., *Anal. Chem.*, **43**, 573 (1971).
- (123) Busch, U., *Chromatographia*, **5**(2-3), 63 (1972).
- (124) Butler, L., Hawkes, S., *J. Chromatogr. Sci.*, **10**, 518 (1972).
- (125) Buvaol, V. V., Levenko, V. A., USSR Patent **283,672** (April 8, 1971).
- (126) Buys, T. S., DeClerk, K., *Anal. Chem.*, **44**, 1273 (1972).
- (127) Buys, T. S., De Clerk, K., *J. Chromatogr. Sci.*, **10**, 722 (1972).
- (128) Buys, T., De Clerk, K., *J. Chromatogr.*, **69**, 87 (1972).
- (129) Caesar, F., *Chromatographia*, **5**, 173 (1972).

- (130) Calam, D., *J. Chromatogr.*, **70**, 146 (1972).
- (131) Carie, G. C., *Anal. Chem.*, **44**, 1905 (1972).
- (132) Carrico, J. P., in "Dynamic Mass Spectrometry," Vol. 3, D. Price, Ed., Heyden and Son Ltd., 1972, pp 1-65.
- (133) Carroll, D. I., Cohen, M. J., Werniund, R. F., U.S. Patent **3,626,180** (Dec. 7, 1971).
- (134) Carruth, G. F., Kobayashi, R., *Anal. Chem.*, **44**, 1047 (1972).
- (135) Castello, G., *J. Chromatogr.*, **66**, 213 (1972).
- (136) Castello, G., D'Amato, G., *ibid.*, **76**, 293 (1973).
- (137) Castello, G., Lunardelli, M., Berg, M., *ibid.*, p 31.
- (138) Chan, S. C., Inel, Y., Tschukow-Roux, E., *Can. J. Chem.*, **50**, 1443 (1972).
- (139) Chaplyghina, Y. K., Moskovtsev, V. V., Molokanov, Y. K., Fursa, O. N., Palamar-chuk, N. A., Golubtsov, S. A., *Khim. Prom.*, **47**, 821 (1971).
- (140) Charrier, G., Dupuis, M. C., *Chromatogra-phia*, **5**, 119 (1972).
- (141) Chastrette, M., Lenhant, G., *J. Chroma-togr.*, **68**, 19 (1972).
- (142) Chatot, G., Murgat, J. P., Grollier, J. F., Fontanges, R., *Method. Phys. Anal.*, **4**, 279 (1968).
- (143) *Chem. Eng. News*, **50**(34), 17 (1972).
- (144) *Chem. Eng. News*, **51**(21), 22 (1973).
- (145) Chen, C. J., Parcher, J. F., *Anal. Chem.*, **43**, 1738 (1971).
- (146) Chen, E., Wentworth, W., *J. Chromatogr.*, **68**, 302 (1972).
- (147) Chesler, S. N., Cram, S. P., *Anal. Chem.*, **43**, 1922 (1971).
- (148) *ibid.*, **44**, 2240 (1972).
- (149) *ibid.*, **45**, 1354 (1973).
- (150) Chesler, S. P., Juvet, R. S., *J. Chromatog. Sci.*, **11**, 320 (1973).
- (151) Chizhkov, V. P., *Zh. Anal. Khim.*, **27**, 1383 (1972).
- (152) Chizhkov, V. P., Usorov, M. I., *Zavod. Lab.*, **37**, 1281 (1971).
- (153) Chizhkov, V. P., Usorov, M. I., Sinitsyna, L. A., *ibid.*, p 1286.
- (154) Choudhary, V. R., Doraiswamy, L. K., *Ind. Eng. Chem., Prod. Res. Develop.*, **10**, 218 (1971).
- (155) Christophe, A., "Sixth International Symposium on Chromatography and Electro-phoresis," Ann Arbor Sci. Publ., Ann Arbor, Mich., 1971, pp 65-72.
- (156) Chumachenko, M. N., Alekseyeva, N. N., *Zh. Anal. Khim.*, **26**, 1805 (1971).
- (157) Chumachenko, M. N., Kolesnik, L. V., *Izv. Akad. Nauk SSSR, Ser. Khim.*, **1971**, 2090.
- (158) Cimbura, G., *J. Chromatogr. Sci.*, **10**, 287 (1972).
- (159) Clementi, S., Savelli, G., Vergoni, M., *Chromatographia*, **5**, 413 (1972).
- (160) Clough, H., Gibb, T. C., Littlewood, A. B., *ibid.*, p 351.
- (161) Cochrane, W., Wilson, B., *J. Chromatogr.*, **63**, 364 (1971).
- (162) Cochrane, W., Wilson, B., Greenhalgh, R., *ibid.*, **75**, 207 (1973).
- (163) Cocks, A., Egger, K., *J. Chem. Soc., Faraday Trans. 1*, **1972**, 423.
- (164) Cohen, M. J., U.S. Patent **3,626,178** (Dec. 7, 1971).
- (165) *ibid.*, **3,626,179** (Dec. 7, 1971).
- (166) *ibid.*, **3,626,182** (Dec. 7, 1971).
- (167) Coleman, A. E., *J. Chromatogr. Sci.*, **11**, 198 (1973).
- (168) Cook, L., Raushel, F., *J. Chromatogr.*, **65**, 556 (1972).
- (169) Cooke, W., *Diss. Abstr.*, **33**(10), 4701B (1973).
- (170) Cope, J. F., *Anal. Chem.*, **45**, 562 (1973).
- (171) Corbin, J., *Diss. Abstr.*, **32**, 785B (1971).
- (172) Cornish, D. W., Grossman, D. M., Jacobs, A. L., Michaelis, A. F., Saisitz, B., *Anal. Chem.*, **45**, 221R (1973).
- (173) Coudert, M., Vergnaud, J., "Sixth Interna-tional Symposium on Chromatography and Electrophoresis," Ann Arbor Sci. Publ., Ann Arbor, Mich., 1971, pp 73-81.
- (174) Coward, R., Smith, P., *J. Chromatogr.*, **61**, 329 (1971).
- (175) Cox, D., Alexander, M., *Appl. Microbiol.*, **25**, 408 (1973).
- (176) Cox, D., Alexander, M., *Bull. Environ. Contam. Toxicol.*, **9**(2), 84 (1973).
- (177) Cram, S. P., Chesler, S. N., *J. Chroma-togr. Sci.*, **11**, 391 (1973).
- (178) Cram, S. P., Juvet, R. S., *Anal. Chem.*, **44**(5), 213R (1972).
- (179) Cramers, C., Rijks, J., Bocek, P., *J. Chromatogr.*, **65**, 29 (1972).
- (180) Craven, D. A., Everett, E. S., Rubel, M., *J. Chromatogr. Sci.*, **9**, 541 (1971).
- (181) Cravey, R. H., Reed, D., Sedgwick, P. R., *Amer. Lab.*, **4**, 63 (1972).
- (182) Crippen, R. C., "Identification of Organic Compounds with the Aid of Gas Chroma-tography," McGraw-Hill, New York, 1973.
- (183) Christophe, A. B., *Chromatographia*, **4**, 455 (1971).
- (184) Cronin, D., *J. Chromatogr.*, **64**, 25 (1972).
- (185) Cronin, D., Gilbert, J., *ibid.*, **71**, 251 (1972).
- (186) Crowne, C. W. P., Harper, M. F., *ibid.*, **61**, 1 (1971).
- (187) Crowne, C. W. P., Harper, M. F., Farrell, P. G., *ibid.*, p 7.
- (188) Cukor, P., Lanning, E. W., *J. Chromatogr. Sci.*, **9**, 487 (1971).
- (189) Cummins, L. M., "Recent Advances in Gas Chromatography," I. I. Domsy and J. A. Perry, Ed., Marcel Dekker, Inc., New York, 1971, pp 313-40.
- (190) Czubryt, J., Gesser, H. D., *J. Chroma-togr.*, **59**, 1 (1971).
- (191) Dagnall, R. M., Fleet, B., Risby, T. H., Deans, D. R., *Anal. Lett.*, **4**, 497 (1971).
- (192) Dagnall, R. M., West, T. S., Whitehead, P., *Anal. Chem.*, **44**, 2074 (1972).
- (193) Dagnell, R. M., Whitehead, P., *Proc. Soc. Anal. Chem.*, **9**, 201 (1972).
- (194) Darlage, L. J., Block, S. S., Weidner, J. P., *J. Chromatogr. Sci.*, **11**, 272 (1973).
- (195) Daughenbaugh, N., *Diss. Abstr.*, **32**(3), 1423B (1971).
- (196) Deans, D. R., *Anal. Chem.*, **43**, 2026 (1971).
- (197) Deans, D. R., *J. Chromatogr. Sci.*, **9**, 729 (1971).
- (198) Deans, D. R., *Proc. Soc. Anal. Chem.*, **9**, 250 (1972).
- (199) Deans, D. R., Huckle, M. T., Peterson, R. M., *Chromatographia*, **4**, 279 (1971).
- (200) Deans, D. R., Scott, I., *Anal. Chem.*, **45**, 1137 (1973).
- (201) Deininger, G., Halasz, I., *Erdol Kohle*, **25**, 394 (1972).
- (202) DeLeenheer, A., *J. Chromatogr.*, **74**, 35 (1972).
- (203) DeLew, R. B., *J. Chromatogr. Sci.*, **10**, 600 (1972).
- (204) DeLigny, C., Van't Verlatt, T., Karthaus, F., *J. Chromatogr.*, **76**, 115 (1973).
- (205) Delventhal, J., Keck, H., Kuchen, W., *ibid.*, **77**, 422 (1973).
- (206) Demayo, A., Comba, M., *Bull. Environ. Contam. Toxicol.*, **8**, 212 (1972).
- (207) Dencker, W. D., Rushneck, D. R., Shoemake, G. R., *Anal. Chem.*, **44**, 1753 (1972).
- (208) Derge, K., *Chromatographia*, **5**(11), 284 (1972).
- (209) *ibid.*, **5**, 415 (1972).
- (210) Derge, K., *J. Chromatogr.*, **77**, 75 (1973).
- (211) Dessey, R. E., Titus, J. A., *Anal. Chem.*, **45**, 124A (1973).
- (212) DeZan, P., Fasanello, J., *J. Chromatogr. Sci.*, **10**, 333 (1972).
- (213) DiCorcia, A., Bruner, F., *J. Chromatogr.*, **62**, 462 (1971).
- (214) DiCorcia, A., Samperi, R., *ibid.*, **77**, 277 (1973).
- (215) Dietz, R. N., Cote, E. A., *Environ. Sci. Technol.*, **7**, 338 (1973).
- (216) Dietz, W. A., *J. Chromatogr. Sci.*, **10**, 423 (1972).
- (217) DiLorenzo, A., *J. Chromatogr.*, **76**, 207 (1973).
- (218) Dimov, N., Petkova, T., *ibid.*, **74**, 165 (1972).
- (219) Dobrochiver, I. G., Zuykina, N. Y., Shish-kin, L. N., Osipov, B. V., *Zavod. Lab.*, **38**, 659 (1972).
- (220) Dolan, J. W., Hall, R. C., Todd, T. M., *J. Ass. Offic. Anal. Chem.*, **55**, 537 (1972).
- (221) Dondi, F., Betti, A., Bighi, C., *J. Chroma-togr.*, **66**, 191 (1972).
- (222) Douglas, A. G., *J. Chromatogr. Sci.*, **9**, 742 (1971).
- (223) *ibid.*, **10**, 425 (1972).
- (224) Downs, H. D., Purcell, J. E., U.S. Patent **3,635,093** (Jan. 18, 1972).
- (225) Dressler, M., Guha, O., Janak, J., *J. Chromatogr.*, **65**, 261 (1972).
- (226) Dressler, M., Martinu, V., Janak, J., *ibid.*, **59**, 429 (1971).
- (227) Dressler, M., Vespalec, R., Janak, J., *ibid.*, p 423.
- (228) Dressman, R. C., *J. Chromatogr. Sci.*, **10**, 468 (1972).
- (229) *ibid.*, p 472.
- (230) Dubsky, H. E., *Chromatographia*, **3**, 366 (1970).
- (231) Dufka, O., Malinsky, J., Vladyka, J., *Chem. Prum.*, **21**, 459 (1971).
- (232) Dunlop, A. S., Pollard, S. A., *Anal. Chem.*, **43**, 1344 (1971).
- (233) Durbin, D. E., *ibid.*, **45**, 818 (1973).
- (234) Dwyer, R. W., *ibid.*, p 1380.
- (235) Eads, E. A., U.S. Patent **3,650,696** (March 21, 1972).
- (236) Ecknig, W., Lenz, E., *J. Chromatogr.*, **64**, 7 (1972).
- (237) Ediz, S., van Swaay, M., McBride, H., *Chem. Instrum.*, **3**, 399 (1972).
- (238) Eggertsen, F. T., Stross, F. H., *Anal. Chem.*, **44**, 709 (1972).
- (239) Eichelberger, J. W., Lichtenberg, J. J., *J. Amer. Water Works Ass.*, **63**, 25 (1971).
- (240) Eisen, O. G., Kiselev, A. V., Pilt, A. E., Rang, S. A., Shcherbakova, K. D., *Chro-matographia*, **4**, 448 (1971).
- (241) Eisen, O. G., Orav, A., Rang, S., *ibid.*, **5**, 229 (1972).
- (242) Eisenberg, F., Jr., in "Analytical Chemistry of Phosphorus Compounds," M. Halmann, Ed., Wiley-Interscience, New York, 1972, pp 69-91.
- (243) Eon, C., Chatterjee, A. K., Karger, B. L., *Chromatographia*, **5**, 28 (1972).
- (244) Eon, C., Pommier, C., Guiochon, G., *ibid.*, **4**, 235 (1971).
- (245) *ibid.*, p 241.
- (246) Evrard, E., *J. Chromatogr.*, **68**, 47 (1972).
- (247) Fanter, D. L., Levy, R. L., Wolf, C. J., *Anal. Chem.*, **44**, 43 (1972).
- (248) Fanter, D. L., Wolf, C. J., *ibid.*, **45**, 565 (1973).
- (249) Farzane, N. G., Ilyasov, L. V., "Automatic Detectors of Gases," *Energia, Shlyuzo-vaya and*, **10**, Moscow M-114, 1972.
- (250) Farzane, N. G., Ilyasov, L. V., *Uch. Zap. Azerb. Inst. Neft. Khim.*, **9**(1), 62 (1970).
- (251) Farzane, N. G., Ilyasov, L. V., Azim-Zade, A. Yu., *Zavod. Lab.*, **38**, 1443 (1972).
- (252) Farzane, N. G., Ilyasov, L. V., Trushkin, Y. V., *Izv. Vyssh. Ucheb. Zaved. Neft Gaz.*, **14**(6), 85 (1971).
- (253) Fatscher, M., Vergnaud, J., *Bull. Soc. Chim. France*, **1971**, 4220.
- (254) Feher, T., Bodrogi, L., *J. Chromatogr.*, **71**, 17 (1972).
- (255) Feltl, L., Smolkova, E., *ibid.*, **65**, 249 (1972).
- (256) Fentiman, A. F., Foltz, R. L., Kinzer, G. W., *Anal. Chem.*, **45**, 580 (1973).
- (257) Fike, W. W., *J. Chromatogr. Sci.*, **11**, 25 (1973).
- (258) Filbert, A. M., "Recent Advances in Gas Chromatography," I. I. Domsy and J. A. Perry, Ed., Marcel Dekker, Inc., New York, 1971, pp 49-98.
- (259) Finkle, B. S., Taylor, D. M., Bonelli, E. J., *J. Chromatogr. Sci.*, **10**, 312 (1972).
- (260) Fishbein, L., "Chromatography of Environ-mental Hazards. Vol. I. Carcinogens, Mu-tagens, and Teratogens," Elsevier, New York, 1972.
- (261) Flath, R. A., "Guide to Modern Methods on Instrumental Analysis," T. H. Gow, Ed., Wiley Interscience, New York, 1972, pp 323-50.
- (262) Fleming, R., Alexander, M., *Appl. Micro-biol.*, **24**, 323 (1972).
- (263) Folmer, O. F., *Anal. Chim. Acta*, **56**, 447 (1971).
- (264) Foon, R., Tait, K., *Trans. Faraday Soc.*, **67**, 3038 (1971).
- (265) Ford, D., Weihman, K., "Recent Advances in Gas Chromatography," I. I. Domsy and J. A. Perry, Ed., Marcel Dekker, Inc., New York, 1971, pp 377-98.
- (266) Fozard, A., Franses, J. J., Wyatt, A. J.,

- Chromatographia*, 5, 130 (1972).
- (267) *Ibid.*, p 377.
- (268) Franc, J., Senkykova, J., *J. Chromatogr.*, **78**, 123 (1973).
- (269) Franc, J., Senkykova, J., Placek, K., *ibid.*, **65**, 197 (1972).
- (270) Franck, A., Orth, H., Bidlingmaier, D., Nussbaum, R., *Chem. Ztg.*, **95**, 219 (1971).
- (271) Franken, J. J., Vidal-Madjar, C., Guiochon, G., *Anal. Chem.*, **43**, 2034 (1971).
- (272) Frankoski, S. P., Siggia, S., *ibid.*, **44**, 507 (1972).
- (273) *Ibid.*, p 2078.
- (274) Frazer, J. W., Carlson, L. R., Kray, A. M., Bertoglio, M. R., Perone, S. P., *ibid.*, **43**, 1479 (1971).
- (275) Freed, D. J., Faulkner, L. R., *ibid.*, **44**, 1194 (1972).
- (276) Freeman, R., *Diss. Abstr.*, **32**, 3876B (1972).
- (277) Freeman, R. B., Wentworth, W. E., *Anal. Chem.*, **43**, 1987 (1971).
- (278) French, M., Jeffries, D., *Bull. Environ. Contam. Toxicol.*, **6**, 460 (1971).
- (279) Fritz, D. F., Kovats, E. sz., *Anal. Chem.*, **45**, 1175 (1973).
- (280) Frycka, J., *J. Chromatogr.*, **65**, 341 (1972).
- (281) *Ibid.*, p 432.
- (282) Frycka, J., Pospisil, J., *ibid.*, **67**, 366 (1972).
- (283) Fujinaga, T., Kuwamoto, T., Murai, S., *Talanta*, **18**, 429 (1971).
- (284) Fuller, E. N., *Anal. Chem.*, **44**, 1747 (1972).
- (285) Gaede, D., Meloan, C. E., *ibid.*, **43**, 1515 (1971).
- (286) Gallegos, E. J., *ibid.*, p 1151.
- (287) Garilli, A., Fabiana, L., Filia, U., Cusi, V., *J. Chromatogr.*, **77**, 3 (1973).
- (288) Garzo, G., Alexander, G., *Chromatographia*, **4**, 554 (1972).
- (289) Gassiot-Matas, M., Julia-Danes, E., *ibid.*, **5**, 493 (1972).
- (290) Gassiot-Matas, M., Pascual-Calveras, J. O., Serra-Marcia, A., *ibid.*, p 328.
- (291) Gerber, J. N., Sawyer, D. T., *Anal. Chem.*, **44**, 1199 (1972).
- (292) Gerezkin, V., *J. Chromatogr.*, **65**, 227 (1972).
- (293) German, A. L., Horning, E. C., *J. Chromatogr. Sci.*, **11**, 76 (1973).
- (294) German, A. L., Pfaffenberger, C. D., The-not, J. P., Horning, M. G., Horning, E. C., *Anal. Chem.*, **45**, 930 (1973).
- (295) Gerritse, R., Huber, J., *J. Chromatogr.*, **71**, 173 (1972).
- (296) Ghosh, S., Sarkar, H., Saha, N., *ibid.*, **74**, 171 (1972).
- (297) Giang, B., *Diss. Abstr.*, **33**, 1039B (1972).
- (298) Gil-Av, E., Schurig, V., *Anal. Chem.*, **43**, 2030 (1971).
- (299) Gill, J. M., Deans, D. R., *J. Chromatogr. Sci.*, **9**, 705 (1971).
- (300) Gill, J. M., *ibid.*, **10**, 1 (1972).
- (301) Gill, J. M., Henselman, J., *Chromatographia*, **5**, 108 (1972).
- (302) Giuffrida, L., "Recent Advances in Gas Chromatography," I. I. Domskey and J. A. Perry, Ed., Marcel Dekker, Inc., New York, 1971, pp 125-35.
- (303) Gleispach, H., Daxenbichler, G., *Chromatographia*, **4**, 400 (1971).
- (304) Giotfelty, D. E., *Anal. Chem.*, **44**, 1250 (1972).
- (305) Gochman, N., Young, D. S., *ibid.*, **45**, 11R (1973).
- (306) Goedert, M., Guiochon, G., *ibid.*, p 1180.
- (307) *Ibid.*, p 1188.
- (308) Golovistikov, Yu. N., *Zavod. Lab.*, **38**, 533 (1972).
- (309) Golovnya, R. V., Arsenyev, Y. N., *Chromatographia*, **4**, 250 (1971).
- (310) Golovnya, R. V., Arsenyev, Y. N., *Izv. Akad. Nauk SSSR. Ser. Khim.*, **1972**, 632.
- (311) Golovnya, R. V., Uralets, V. P., *ibid.*, p 1982.
- (312) Goma, G., Durant, G., *Water Res.*, **5**, 545 (1971).
- (313) Goodfellow, G., Webber, H., *Chem. Ind. (London)*, **1972**, 127.
- (314) Gordon, A., Frigerio, A., *J. Chromatogr.*, **73**, 401 (1972).
- (315) Goretti, G., Possanzini, M., *ibid.*, **77**, 317 (1973).
- (316) Gough, A., Simpson, C., *ibid.*, **68**, 31 (1972).
- (317) Gough, T., Webb, K., *ibid.*, **64**, 201 (1972).
- (318) Grant, D. W., *J. Chromatogr. Sci.*, **10**, 705 (1972).
- (319) Grant, D. W., Clarke, A., *Anal. Chem.*, **43**, 1951 (1971).
- (320) Grayson, M. A., Bellina, J. J., *ibid.*, **45**, 487 (1973).
- (321) Grayson, M. A., Levy, R. L., Wolf, C. J., *ibid.*, p 373.
- (322) Greco, M., Marranci, G., *J. Chromatogr.*, **77**, 91 (1973).
- (323) Greenhalgh, R., Cochrane, W., *ibid.*, **70**, 37 (1972).
- (324) Greer, D. G., Bydalek, T. J., *Environ. Sci. Technol.*, **7**, 153 (1973).
- (325) Griffin, G. W., Dzidic, I., Carroll, D. I., Stilwell, R. N., Horning, E. C., *Anal. Chem.*, **45**, 1204 (1973).
- (326) Grimmer, G., Hildebrandt, A., Bohnke, H., *Erdol Kohle*, **25**, 442 (1972).
- (327) *Ibid.*, p 531.
- (328) Grob, K., Grob, G., *Chromatographia*, **5**, 3 (1972).
- (329) Grob, R. L., McGonigle, E. J., *J. Chromatogr.*, **59**, 13 (1971).
- (330) Grobler, A., *J. Chromatogr. Sci.*, **10**, 128 (1972).
- (331) Grobler, A., Kada, T., Sarosi, G., "Proc. Third Anal. Chem. Conf.," Budapest, Vol. 1, 1970, pp 223-26.
- (332) Grohmann, K., Parr, W., *Chromatographia*, **5**, 18 (1972).
- (333) Grubner, O., *Anal. Chem.*, **43**, 1934 (1971).
- (334) Grubner, O., Goldin, A. S., *ibid.*, **45**, 944 (1973).
- (335) Grubner, O., Underhill, D., *J. Chromatogr.*, **73**, 1 (1972).
- (336) Grushka, E., *Anal. Chem.*, **44**, 1733 (1972).
- (337) Grushka, E., *Chem. Technol.*, **1971**, 745.
- (338) Grushka, E., Guiochon, G., *J. Chromatogr. Sci.*, **10**, 649 (1972).
- (339) Grushka, E., Baraya, M. Y., Cooke, W. D., *ibid.*, **9**, 653 (1971).
- (340) Grushka, E., Guiochon, G., *ibid.*, **10**, 649 (1972).
- (341) Grushka, E., Lo, F. B., *Anal. Chem.*, **45**, 903 (1973).
- (342) Grushka, E., Monacelli, G. C., *ibid.*, **44**, 484 (1972).
- (343) Grushka, E., Scott, R. P. W., *ibid.*, **45**, 1626 (1973).
- (344) Grushka, E., Solsky, J. F., *ibid.*, p 1836.
- (345) Guenther, E., Gilbertson, G., Koenig, R. T., *ibid.*, p 45R.
- (346) Guerin, M. R., *Anal. Lett.*, **4**, 751 (1971).
- (347) Guglia, V. G., Golden, A. D., Zhukhovitskii, A. A., Kaimanovskii, V. I., Roitberg, M. B., Rabinovich, A. Z., USSR Patent **276,499** (Oct. 15, 1970).
- (348) Guglia, V. G., Kaimanovskii, V. I., Shepeliev, V. Ye., Zhukhovitskii, A. A., *Zavod. Lab.*, **38**, 535 (1972).
- (349) Guha, O., Janak, J., *J. Chromatogr.*, **68**, 325 (1972).
- (350) Guillemin, C. L., Deleuil, M., Cirendini, S., Vermont, J., *Anal. Chem.*, **43**, 2015 (1971).
- (351) Guillemin, C. L., LePage, M., de Vries, A. J., *J. Chromatogr. Sci.*, **9**, 470 (1971).
- (352) Guiochon, G., *Chromatographia*, **4**, 404 (1971).
- (353) Guiochon, G., *J. Chromatogr. Sci.*, **9**, 512 (1971).
- (354) Guiochon, G., Jacob, L., *Usp. Khromatogr.*, **1972**, pp 170-78.
- (355) Gunzler, H., *Erdol Kohle*, **24**, 633 (1971).
- (356) Guthrie, F. A., McMasters, D. L., Ed., "Directory of Members and Divisional History," ACS Division of Analytical Chemistry, 1973.
- (357) Gutsche, B., Herrmann, R., Ruediger, K., *Fresenius' Z. Anal. Chem.*, **258**, 273 (1972).
- (358) Hachenberg, H., *Erdol Kohle*, **24**, 630 (1971).
- (359) Haddadin, J., Stirland, R., Preston, N., Collard, P., *Appl. Microbiol.*, **25**, 40 (1973).
- (360) Haken, J. K., *J. Chromatogr.*, **73**, 419 (1972).
- (361) Haken, J. K., *J. Chromatogr. Sci.*, **11**, 144 (1973).
- (362) Haken, J. K., Ho, D. K. M., Withers, M. K., *ibid.*, **10**, 566 (1972).
- (363) Haken, J. K., McKay, T. R., *Anal. Chem.*, **45**, 1251 (1973).
- (364) Hammar, C., Hesslering, R., *ibid.*, **43**, 298 (1971).
- (365) Hansen, N. H., Kines, K., Nielsen, T., *J. Chromatogr. Sci.*, **9**, 631 (1971).
- (366) Harris, R. J., Sr., Harris, R. J., Jr., U.S. Patent **3,641,822** (Feb. 15, 1972).
- (367) *Ibid.*, **3,641,823** (Feb. 15, 1972).
- (368) Harris, W. E., *J. Chromatogr. Sci.*, **11**, 184 (1973).
- (369) Hartkopf, A., *ibid.*, **10**, 145 (1972).
- (370) Hartmann, C. H., *Anal. Chem.*, **45**, 733 (1973).
- (371) Hartmann, C. H., U.S. Patent **3,607,096** (Sept. 21, 1971).
- (372) Hartung, H. A., Dwyer, R. W., *Anal. Chem.*, **44**, 1743 (1972).
- (373) Haruki, T., Hayashi, Y., U.S. Patent **3,626,761** (Dec. 14, 1971).
- (374) Hasson, J. C., Kulfarni, M. V., *Anal. Chem.*, **44**, 1586 (1972).
- (375) Hastings, C., Aue, W., Larsen, F., *J. Chromatogr.*, **60**, 329 (1971).
- (376) Hattman, E. A., Schultz, H., Ortuglio, C., *Anal. Chem.*, **45**, 345R (1973).
- (377) Hauser, T. R., Pattison, J. N., *Environ. Sci. Technol.*, **6**, 549 (1972).
- (378) Haverkamp, J., Kamerling, J. P., Vligenthart, J. F. G., *J. Chromatogr.*, **59**, 281 (1971).
- (379) Hawkes, S. J., "Recent Advances in Gas Chromatography," I. I. Domskey and J. A. Perry, Ed., Marcel Dekker, Inc., New York, 1971, pp 13-47.
- (380) Hegedus, L. L., Petersen, E. E., *J. Chromatogr. Sci.*, **9**, 551 (1971).
- (381) Hems, R. V., Adams, J. B., *Lab. Practice*, **21**, 430 (1972).
- (382) Henderson, W., Steel, G., *Anal. Chem.*, **44**, 2302 (1972).
- (383) Henly, R. S., *J. Chromatogr. Sci.*, **11**, 221 (1973).
- (384) Henneberg, D., Casper, K., Ziegler, E., *Chromatographia*, **5**, 209 (1972).
- (385) Hertl, W., Neumann, M., *J. Chromatogr.*, **60**, 319 (1971).
- (386) Hettinger, J. D., Hubbard, J. R., Gill, J. M., Miller, L. A., *J. Chromatogr. Sci.*, **9**, 710 (1971).
- (387) Hey, H., *Fresenius' Z. Anal. Chem.*, **256**, 361 (1971).
- (388) Hill, H. H., Aue, W. A., *J. Chromatogr.*, **74**, 311 (1972).
- (389) Hishta, C., Mays, D. L., Grafoalo, M., *Anal. Chem.*, **43**, 1530 (1971).
- (390) Hites, R. A., Biemann, K., *Science*, **178**, 160 (1972).
- (391) Hobrecht, H. D., Klimowski, R. J., *Chromatogr. Newslett.*, **1** (1), 23 (1972).
- (392) Hoffmann, J., Tilgner, H., *Z. Physik. Chem.*, **246**, 407 (1971).
- (393) Hoffsommer, J. C., *J. Chromatogr.*, **51**, 243 (1970).
- (394) Holden, A. V., *Proc. Soc. Anal. Chem.*, **10**, 31 (1973).
- (395) Holland, J. F., Sweeley, C. C., Thrush, R. E., Teets, R. E., Bieber, M. A., *Anal. Chem.*, **45**, 308 (1973).
- (396) Hollowell, C. D., Gee, G. Y., McLaughlin, R. D., *ibid.*, p 63A.
- (397) Horning, E. C., Horning, M. G., "Recent Advances in Gas Chromatography," I. I. Domskey and J. A. Perry, Ed., Marcel Dekker, Inc., New York, 1971, pp 34-75.
- (398) Horning, E. C., Horning, M. G., Carroll, D. I., Dzidic, I., Stilwell, R. N., *Anal. Chem.*, **45**, 936 (1973).
- (399) Horton, A. D., *J. Chromatogr. Sci.*, **10**, 125 (1972).
- (400) Hoshikawa, Y., Koike, K., Kuriyama, T., *Bunseki Kagaku*, **21**, 307 (1972).
- (401) Hrdina, J., U.S. Patent **3,604,268** (Sept. 14, 1971).
- (402) Huang, T., Yang, F., Huang, C., Kuo, C., *J. Chromatogr.*, **70**, 13 (1972).
- (403) Hukhovitskii, A. A., Sazonov, M. L., Lunskii, M. K., Vyatkin, A. F., *Zh. Anal. Khim.*, **27**, 748 (1972).
- (404) Hussey, C. L., Parcher, J. F., *Anal. Chem.*, **45**, 926 (1973).
- (405) Iacconi, P., Renucci, G., *Chim. Anal. (Paris)*, **53**, 327 (1971).

- (406) Ilkova, E. L., Mistryukov, E. A., *J. Chromatogr. Sci.*, **9**, 569 (1971).
- (407) *Ind. Res.*, **14**(6) C1, C4 (1972).
- (408) *Ind. Res.*, **14**(6), 41 (1972).
- (409) Inoue, M., Tsuda, T., Ito, T., Ishii, D., *Kogyo Kagaku Zasshi*, **74**, 880 (1971).
- (410) Isbell, A., *Diss. Abstr.*, **32**, 2046B (1971).
- (411) Ishii, D., Tsuda, T., Tokoro, N., *Bunseki Kagaku*, **21**, 363 (1972).
- (412) *Ibid.*, p 367.
- (413) Ivanov, G. A., Guglia, V. G., Zhukhovitskii, A. A., *Zavod. Lab.*, **39**, 15 (1973).
- (414) Iwata, Y., Westlake, W. E., Gunther, F. A., *Bull. Environ. Contam. Toxicol.*, **9**, 204 (1973).
- (415) Jackson, H. W., *J. Chromatogr. Sci.*, **9**, 706 (1971).
- (416) Jacquilot, P., Thomas, G., *J. Chromatogr.*, **66**, 121 (1972).
- (417) Jain, N. C., Cravey, R. H., *J. Chromatogr. Sci.*, **10**, 263 (1972).
- (418) Jakubiec, R. J., Metcalfe, L. D., Boer, B., Wang, C. N., Japan Oil Chem. Soc.-Amer. Oil Chemists' Soc. Joint Meeting, Los Angeles, 1972.
- (419) Janghorbani, M., Freund, H., *Anal. Chem.*, **45**, 325 (1973).
- (420) Janik, A., *Chem. Anal. (Warsaw)*, **18**, 29 (1973).
- (421) Janak, J., *Usp. Khromatogr.*, **1972**, 268-93.
- (422) Janak, J., Martinu, V., Ruzickova, J., *J. Chromatogr.*, **78**, 127 (1973).
- (423) *J. Chromatogr. Sci.*, **10**, 352 (1972).
- (424) *Ibid.*, (2), G2-52 (1972).
- (425) *Ibid.*, **11**(1), G2-54 (1973).
- (426) Jeffery, P. G., Kipping, P. J., "Gas Analysis by Gas Chromatography," 2nd ed., Pergamon Press, N.Y., 1972.
- (427) Jellum, E., Stokke, O., Eldjarn, L., *Anal. Chem.*, **45**, 1099 (1973).
- (428) Jeltens, R., Tonkelaar, W. D., *Water Res.*, **6**, 271 (1972).
- (429) Jenden, D. J., Roch, M., Booth, R., *J. Chromatogr. Sci.*, **10**, 151 (1972).
- (430) Jentoft, R. E., Gouw, T. H., *Anal. Chem.*, **44**, 681 (1972).
- (431) Jernejcic, M., Kidric-Zajas, J., Kremser, M., Premru, L., *J. Chromatogr.*, **64**, 371 (1972).
- (432) Johns, T., Stapp, C., *J. Chromatogr. Sci.*, **11**, 234 (1973).
- (433) Johnson, R. P., Price, S. J. W., *Can. J. Chem.*, **50**, 50 (1972).
- (434) Johnston, A. E., Dutton, H. J., *J. Amer. Oil Chem. Soc.*, **49**, 98 (1972).
- (435) Jones, P., Nickless, G., *J. Chromatogr.*, **73**, 19 (1972).
- (436) *Ibid.*, **76**, 285 (1973).
- (437) Juvet, R. S., Smith, J. L. S., Li, K. P., *Anal. Chem.*, **44**, 49 (1972).
- (438) Kagawa, S., Fujita, K., Tada, K., Nakamori, I., *ibid.*, p 1540.
- (439) Kaiser, R. E., *ibid.*, **45**, 965 (1973).
- (440) Kaiser, R., *Chromatographia*, **5**, 177 (1972).
- (441) Kaiser, R., "Preparative Gas Chromatography," A. Zlatikis and Pretorius, Ed., Wiley Interscience, New York, 1971, pp 187-210.
- (442) Kaiser, R., *Usp. Khromatogr.*, **1972**, 193-214.
- (443) Kalaschnikova, E. V., Kiselev, A. V., Petrova, R. S., Shcherbakova, K. D., *Chromatographia*, **4**, 495 (1971).
- (444) Kaminski, E., Wasowicz, E., Przybylski, R., *Chem. Anal. (Warsaw)*, **17**, 1307 (1972).
- (445) Kamps, L. R., McMahon, B., *J. Ass. Offic. Anal. Chem.*, **55**, 590 (1972).
- (446) Kananen, G., Osiewicz, R., Sunshine, I., *J. Chromatogr. Sci.*, **10**, 283 (1972).
- (447) Kane, D. M., Karasek, F. W., *ibid.*, p 501.
- (448) Kaplar, L., Czita, C., Takacs, J., Tarjan, G., *J. Chromatogr.*, **65**, 115 (1972).
- (449) Karasek, F. W., *Anal. Chem.*, **43**, 1982 (1971).
- (450) *Ibid.*, **44**(4), 32A (1972).
- (451) Karasek, F. W., Cohen, M. J., Carroll, D. I., *J. Chromatogr. Sci.*, **9**, 390 (1971).
- (452) Karasek, F. W., Kane, D. M., *Anal. Chem.*, **45**, 576 (1973).
- (453) Karasek, F. W., Kane, D. M., *J. Chromatogr. Sci.*, **10**, 673 (1972).
- (454) Karasek, F. W., Keller, R. A., *ibid.*, p 626.
- (455) Karasek, F. W., Smythe, R. J., *Anal. Chem.*, **43**, 2008 (1971).
- (456) Karasek, F. W., Tatone, O. S., Kane, D. M., *ibid.*, **45**, 1210 (1973).
- (457) Karger, B. L., Sibley, E., *ibid.*, p 740.
- (458) Karger, B. L., Snyder, L. R., Horvath, C., "An Introduction to Separation Science," Interscience-Wiley, New York, 1973.
- (459) Karlens, J., *J. Chromatogr. Sci.*, **10**, 642 (1972).
- (460) Karmen, A., Haut, H., *Anal. Chem.*, **45**, 822 (1973).
- (461) Karmen, A., Kelly, E. L., *ibid.*, **43**, 1992 (1971).
- (462) Kauffmann, H., Knapp, O., Straub, H., *Chromatographia*, **5**, 558 (1972).
- (463) Kawabata, T., Matsui, M., Ishibashi, T., Nakamura, M., *Bunseki Kagaku*, **21**, 1326 (1972).
- (464) Kawahara, F. K., *J. Chromatogr. Sci.*, **10**, 629 (1972).
- (465) Keiko, V. V., Prokopiev, B. V., Kuzmenko, L. P., Kalinina, N. A., Modonov, V. B., *Izv. Akad. Nauk SSSR, Ser. Khim.*, **1972**, 2697.
- (466) Keiler, H., Scheurle, B., Sabel, J., Hainz, J., Winterscheidt, H., *Mol. Cryst. Liq. Cryst.*, **12**, 113 (1971).
- (467) Keller, J. S., Veening, H., Willeford, B. R., *Anal. Chem.*, **43**, 1516 (1971).
- (468) Keller, R. A., *J. Chromatogr. Sci.*, **11**, 49 (1973).
- (469) *Ibid.*, p 188.
- (470) *Ibid.*, p 223.
- (471) Kelly, P. C., Harris, W. E., *Anal. Chem.*, **43**, 1170 (1971).
- (472) *Ibid.*, p 1184.
- (473) Kelly, R. J., *J. Chromatogr.*, **71**, 337 (1972).
- (474) Kelsey, M., Keskiner, A., Moscatelli, E., *ibid.*, **75**, 294 (1973).
- (475) *Khim. Uzbek. Zh.*, **15**(6), 28 (1971).
- (476) Khokhlov, A. N., Okhotnikov, B. P., Zhukovitskii, A. A., *Zavod. Lab.*, **37**, 533 (1971).
- (477) Khoury, F., Robinson, D. B., *J. Chromatogr. Sci.*, **10**, 683 (1972).
- (478) Kikot, V. V., Safronova, L. N., *Zavod. Lab.*, **38**, 161 (1972).
- (479) Killer, F. C. A., *Proc. Soc. Anal. Chem.*, **9**(3), 71 (1972).
- (480) King, R. W., *Anal. Chem.*, **45**, 169R (1973).
- (481) Kiselev, A. V., Lomonosov, M. V., "Aspects in Gas Chromatography," H. G. Struppe, Ed., Akad. Verlag, Berlin, 1971, pp 48-66.
- (482) Kiselev, A. V., *Usp. Khromatogr.*, **1972**, 33-64.
- (483) Kiselev, A. V., Zung, L., Nikitin, Yu. S., *Kolloid. Zh.*, **33**, 224 (1971).
- (484) Kishimoto, K., *Bunseki Kagaku*, **21**, 1643 (1972).
- (485) Kishimoto, K., Miyauchi, H., Musha, S., *ibid.*, p 852.
- (486) Kishimoto, K., Miyauchi, H., Musha, S., *J. Chromatogr. Sci.*, **10**, 220 (1972).
- (487) Kishimoto, K., Musha, S., *ibid.*, **9**, 608 (1971).
- (488) Klatt, L., Carr, P., Krusberg, R. J., *Chem. Instrum.*, **3**, 327 (1972).
- (489) Klein, P. D., Haumann, J. R., Eisler, W. J., *Anal. Chem.*, **44**, 490 (1972).
- (490) Kleopfer, R. D., Fairless, B. J., *Environ. Sci. Technol.*, **6**, 1036 (1972).
- (491) Klopfenstein, C. E., *J. Chromatogr. Sci.*, **10**, 22 (1972).
- (492) Knox, J. H., Saleem, M., *ibid.*, p 80.
- (493) Ko, E. C. F., Leftek, K. T., *Can. J. Chem.*, **50**, 1297 (1972).
- (494) Kojima, T., Morishita, F., *Bunseki Kagaku*, **20**, 1303 (1971).
- (495) Kolesnikova, L. P., "Gas Chromatography in Investigations of Natural Gases, Petroleum, and Condensates," Nedra, Moscow, 1972.
- (496) Kolloff, R. H., Greenhaus, H. L., Arms, R. A., U.S. Patent 3,621,707 (Nov. 23, 1971).
- (497) Komanova, E., Knopova, V., Koman, V., *J. Chromatogr. Sci.*, **73**, 231 (1972).
- (498) Konig, H., *Fresenius' Z. Anal. Chem.*, **255**, 123 (1971).
- (499) Korol, A. N., *Usp. Khim.*, **41**, 321 (1972).
- (500) Korol, A. N., *Zh. Anal. Khim.*, **27**, 209 (1972).
- (501) Koslow, S. H., Cattabeni, F., Costa, E., *Science*, **176**, 177 (1972).
- (502) Kozeiko, T. A., Mashkevich, A. I., *Zavod. Lab.*, **39**, 25 (1973).
- (503) Krajnovic, I., Osterman, D., *Nafta (Zagreb)*, **22**(3), 155 (1971).
- (504) Kraus, G., Seifert, K., Schubert, H., *Z. Chem.*, **11**, 428 (1971).
- (505) Kraus, G., Seifert, K., Zschke, H., Schubert, H., *ibid.*, p 22.
- (506) Krichmar, S. I., Stepanienko, V. Y., Galun, T. M., *Zh. Anal. Khim.*, **26**, 1340 (1971).
- (507) Kruglova, O. A., Petrov, B. I., Bortnikov, G. N., Vyazankin, N. S., *Izv. Akad. Nauk SSSR, Ser. Khim.*, **1971**, 2242.
- (508) Krupcik, J., Liska, O., Nemecek, J., Duchesne, Y. P., Tesarik, K., *Chromatographia*, **5**, 252 (1972).
- (509) Krylov, B. K., Yashin, Ya. I., *Khim. Prom.*, **48**, 349 (1972).
- (510) Kuznietsova, N. P., Fedorov, V. A., Yushkin, A. A., *Zh. Anal. Khim.*, **27**, 821 (1972).
- (511) Laatikainen, T., Peltonen, J., Nylander, P., *Steroids*, **21**, 347 (1973).
- (512) Landis, W. R., Alford, W. C., Perrine, T. D., Thomas, G. M., Anderson, F. O., *Appl. Spectrosc.*, **25**(1), 44 (1971).
- (513) Landowne, R. A., Morosani, R. W., Herrmann, R. A., King, R. M., Schmus, H. G., *Anal. Chem.*, **44**, 1961 (1972).
- (514) Langer, S. H., *ibid.*, p 1915.
- (515) Lao, R. C., Thomas, R. S., Oja, H., Dubois, L., *ibid.*, **45**, 908 (1973).
- (516) Lasa, J., Korus, A., Kilarska, M., *Inst. Nucl. Phys., Cracow Rep. No. 738*, **1970**, p 16.
- (517) Leary, J. J., Justice, J. B., Tsuge, S., Lowry, S. R., Isenhour, T. L., *J. Chromatogr. Sci.*, **11**, 201 (1973).
- (518) Lebediev, V. V., Molodyk, A. D., Morozova, L. N., Korenchuk, N. M., *Zavod. Lab.*, **38**, 525 (1972).
- (519) Leenheer, A., *J. Chromatogr.*, **74**, 35 (1972).
- (520) Lenoir, J. Y., Rojey, A., *Separ. Sci.*, **7**, 13 (1972).
- (521) Lephardt, J. O., *Diss. Abstr.*, **33**, 1482B (1972).
- (522) Lephardt, J. O., Bulkin, B. J., *Anal. Chem.*, **45**, 706 (1973).
- (523) Letterer, R., *Glas-Instr.-Tech.*, **15**, 1234 (1971).
- (524) Levy, R. L., Fanter, D. L., Wolf, C. J., *Anal. Chem.*, **44**, 38 (1972).
- (525) Levy, R. L., Grayson, M. A., *J. Chromatogr. Sci.*, **9**, 687 (1971).
- (526) Lew, H. Y., *J. Amer. Oil Chem. Soc.*, **49**, 665 (1972).
- (527) Liao, H. L., Martire, D. E., *Anal. Chem.*, **44**, 498 (1972).
- (528) Liebich, H. M., Douglas, D. R., Zlatkis, A., Muggler-Chavan, F., *J. Agr. Food Chem.*, **20**, 96 (1972).
- (529) Liebman, S. A., Ahlstrom, D. H., Nauman, C. D., Averitt, R., Walker, J. L., Levy, E. J., *Anal. Chem.*, **45**, 1360 (1973).
- (530) Ligny, C. D., Van't Verliatt, T., Karthaus, F., *J. Chromatogr.*, **76**, 115 (1973).
- (531) Lino, L. L., *Amer. Lab.*, **5**(2), 71 (1973).
- (532) Liparskii, V. N., Berezkin, V. G., *Zavod. Lab.*, **38**, 657 (1972).
- (533) Litchfield, C., "Analysis of Triglycerides," Academic Press, New York, 1972.
- (534) Littlewood, A. B., "Aspects in Gas Chromatography," H. G. Struppe, Ed., Akad. Verlag, Berlin, Germany, 1971, pp 81-95.
- (535) Lloyd, J. B. F., *Analyst (London)*, **97**, 708 (1972).
- (536) Locke, D. C., *J. Chromatogr. Sci.*, **11**, 120 (1973).
- (537) Locke, D. C., Schermund, J. T., Banner, B., *Anal. Chem.*, **44**, 90 (1972).
- (538) Loda, R. T., Sethi, D. S., *Anal. Lett.*, **4**, 709 (1971).
- (539) Longbottom, J. E., *Anal. Chem.*, **44**, 1111 (1972).
- (540) Longbottom, J. E., *J. Chromatogr. Sci.*, **11**, 13 (1973).
- (541) Lorenz, L. J., *Diss. Abstr.*, **32**, 4458B (1972).
- (542) Lorenz, L. J., Rogers, L. B., *Anal. Chem.*, **43**, 1593 (1971).
- (543) Louis, R., *Erdol Kohle*, **24**, 88 (1971).
- (544) *Ibid.*, **25**, 582 (1972).
- (545) Lovelock, J. E., Maggs, R. J., Adlard, E. R., *Anal. Chem.*, **43**, 1962 (1971).

- (546) Lovelock, J. E., Simmonds, P. G., Shoemaker, G. R., *ibid.*, p 1958.
- (547) Low, M. J. D., *J. Agr. Food Chem.*, **19**, 1124 (1971).
- (548) Lubkowitz, J., Parker, W., *J. Chromatogr.*, **62**, 53 (1971).
- (549) Lubkowitz, J., Montoloy, D., Parker, W., *ibid.*, **76**, 21 (1973).
- (550) Lucero, D. P., *J. Chromatogr. Sci.*, **10**, 463 (1972).
- (551) Lucero, D. P., Smith, P. H., *ibid.*, p 544.
- (552) Luisetti, R. U., Yunes, R. A., *ibid.*, **9**, 624 (1971).
- (553) Lukac, S., Sevcik, J., *Chromatographia*, **5**, 258 (1972).
- (554) *ibid.*, p 311.
- (555) Lunska, M. K., Vyatkin, A. F., Sazonov, M. L., Zhukhovitskii, A. A., *Zavod. Lab.*, **37**, 1290 (1971).
- (556) Lysyj, T., Newton, P. R., Taylor, W. J., *Anal. Chem.*, **43**, 1277 (1971).
- (557) MacDonald, W. R., Habgood, H. W., "21st Canadian Chem. Eng. Conference," Montreal, 1971.
- (558) MacLeod, N., *Chromatographia*, **5**, 516 (1972).
- (559) Macnaughton, D., Rogers, L. B., Wernimont, G., *Anal. Chem.*, **44**, 1421 (1972).
- (560) Maggs, R. J., Joynes, P. L., Davies, A. J., Lovelock, J. E., *ibid.*, **43**, 1966 (1971).
- (561) Malan, E., Brink, B., *Chromatographia*, **5**, 182 (1972).
- (562) Malan, E., Loubser, N. H., Boshoff, A. S. J., Els, O., *ibid.*, **4**, 475 (1971).
- (563) Mallik, K. L., Kuchhal, R. K., *Chem. Ind. (London)*, **1972**, 260.
- (564) Manara, G., Taramasso, M., *J. Chromatogr.*, **65**, 349 (1972).
- (565) *ibid.*, **77**, 109 (1973).
- (566) Mann, J. R., Preston, S. T., Jr., *J. Chromatogr. Sci.*, **11**, 216 (1973).
- (567) Markey, S. P., Simons, S. L., *Anal. Chem.*, **45**, 818 (1973).
- (568) Martin, S., Rowland, M., *J. Pharm. Soc.*, **61**, 1235 (1972).
- (569) Massart, D. L., Janssens, C., Kaufman, L., Smits, R., *Anal. Chem.*, **44**, 2390 (1972).
- (570) Master, R. C., *J. Amer. Oil Chem. Soc.*, **48**, 191 (1970).
- (571) Matsumoto, H., Futami, H., Morita, F., Morita, Y., *Bull. Chem. Soc. Japan*, **44**, 3170 (1971).
- (572) Maynard, V., Grushka, E., *Anal. Chem.*, **44**, 1427 (1972).
- (573) McAllister, W. A., Southerland, W. V., *ibid.*, **43**, 1536 (1971).
- (574) McClure, V., *J. Chromatogr.*, **70**, 168 (1972).
- (575) McCoy, R., *Diss. Abstr.*, **32**, 1410B (1971).
- (576) McDonald, K. L., *Anal. Chem.*, **44**, 1298 (1972).
- (577) McFadden, W., "Techniques of Combined Gas Chromatography/Mass Spectrometry: Applications in Organic Analysis," Wiley-Interscience, New York, 1973.
- (578) McLean, W. R., Stanton, D. L., Penketh, G. E., *Proc. Soc. Anal. Chem.*, **9**, 296 (1972).
- (579) McLeod, H. A., Wales, P. J., *J. Agr. Food Chem.*, **20**, 624 (1972).
- (580) McNair, H. M., Chandler, C. D., *J. Chromatogr. Sci.*, **11**, 454 (1973).
- (581) McNair, H. M., Cooke, W. M., *Amer. Lab.*, **5**(2), 12 (1973).
- (582) McNair, H. M., Cooke, W. M., *J. Chromatogr. Sci.*, **10**, 27 (1972).
- (583) Meijer, G. M., Ritter, F. J., Persoons, C. J., Minks, A. K., Voerman, S., *Science (London)*, **175**, 1469 (1972).
- (584) Melnichenko, L. S., Zemlyanskii, N. N., Chernoplekova, V. A., Kocheshkov, K. A., *Izv. Akad. Nauk SSSR. Ser. Khim.*, **1972**, 1384.
- (585) Menger, F. M., Epstein, G. A., Goldberg, D. A., Reiner, E., *Anal. Chem.*, **44**, 423 (1972).
- (586) Merkus, F. W. H. M., Jaspers-vanWouw, M. G. J., Roovers-Bollen, J. F. C., *Pharm. Weekblad.*, **107**(7), 98 (1972).
- (587) Merritt, C., Jr., Robertson, D. H., *Anal. Chem.*, **44**, 60 (1972).
- (588) Mertens, M. T., Evrard, E., "Sixth International Symposium on Chromatography and Electrophoresis," Ann Arbor Sci. Publ., Ann Arbor, 1971, pp 114-17.
- (589) Metzow, W., Fell, B., *Erdol Kohle*, **25**, 311 (1972).
- (590) Metzger, H. D., Radszweit, K. G., *Chromatographia*, **5**, 186 (1972).
- (591) Meuzelaar, H. L. C., Veld, R. A., *J. Chromatogr. Sci.*, **10**, 213 (1972).
- (592) Middleditch, B. S., Desiderio, D. M., *Anal. Chem.*, **45**, 806 (1973).
- (593) Mieure, J. P., Converse, J. G., Dietrich, M. W., Fowler, L., *ibid.*, **44**, 1332 (1972).
- (594) Mignano, M. J., Rony, P. R., Grenoble, D., Purcell, J. E., *J. Chromatogr. Sci.*, **10**, 637 (1972).
- (595) Mir, L., *ibid.*, **9**, 436 (1971).
- (596) Mitchell, J., *Diss. Abstr.*, **32**, 788B (1971).
- (597) Mitchell, J., Jr., Chiu, J., *Anal. Chem.*, **45**, 273R (1973).
- (598) Mitooka, M., *Bunseki Kagaku*, **20**, 1542 (1971).
- (599) *ibid.*, **21**, 189 (1972).
- (600) *ibid.*, p 197.
- (601) *ibid.*, p 354.
- (602) *ibid.*, p 614.
- (603) *ibid.*, p 717.
- (604) *ibid.*, p 729.
- (605) *ibid.*, p 867.
- (606) *ibid.*, p 1043.
- (607) *ibid.*, p 1242.
- (608) *ibid.*, p 1437.
- (609) *ibid.*, p 1447.
- (610) Moffatt, A., Horning, E., Martin, S., Rowland, M., *J. Chromatogr.*, **66**, 255 (1972).
- (611) Molnar, E., Mertz, P., Takacs, J., *ibid.*, p 205.
- (612) Molodyk, A. D., Bondar, G. V., Morozova, L. N., *Zavod. Lab.*, **38**, 129 (1972).
- (613) Mon, T. R., *Res. Develop.*, **22**(12), 14 (1971).
- (614) Monfort, J. P., *Chim. Anal. (Paris)*, **53**, 646 (1971).
- (615) Mori, Y., *J. Chromatogr.*, **66**, 9 (1972).
- (616) *ibid.*, **70**, 31 (1972).
- (617) Moro-oka, Y., *Trans. Faraday Soc.*, **67**, 3381 (1971).
- (618) Morozova, O. Y., Zemskova, Z. K., Petrov, A. A., *Neftekhimiya*, **12**, 635 (1972).
- (619) Moseman, R., *Diss. Abstr.*, **33**, 90B (1972).
- (620) Moseman, R., Aue, W., *J. Chromatogr.*, **63**, 229 (1971).
- (621) Mueller, P. K., Kothny, E. L., *Anal. Chem.*, **45**, 1R (1973).
- (622) Mule, S. J., *J. Chromatogr. Sci.*, **10**, 275 (1972).
- (623) Mutsaers, P. M., Van Steen, J. E., *J. Inst. Petrol.*, **1972**, 102.
- (624) Myers, A., Smith, R. N. L., *Chromatographia*, **5**, 521 (1972).
- (625) Nakamura, G. R., Noguchi, T., Jackson, D., Banks, D., *Anal. Chem.*, **44**, 408 (1972).
- (626) Narain, C., *Proc. Soc. Anal. Chem.*, **8**(2), 40 (1971).
- (627) Narmetova, G., Ryabova, N. D., *Uzbek. Khim. Zh.*, **1971**, 40.
- (628) *NASA Tech. Brief*, **B72-10249**, 1972.
- (629) Nazarova, L. I., Lulova, N. I., Nikanorov, Y. M., Kuzmina, A. V., Kotelnikov, B. P., Bespalov, Y. I., *Khim. Tekhnol. Topliv. Masel.*, **16**, 51 (1971).
- (630) Nazarova, L. I., Siriuk, A. G., Nikanorov, Y. M., Makeyeva, Y. D., *ibid.*, **17**, 23 (1972).
- (631) Necasova, M., Tesarik, K., *J. Chromatogr.*, **79**, 15 (1973).
- (632) Neff, W. E., Pryde, E. H., Selke, E., Cowan, J. C., *J. Chromatogr. Sci.*, **10**, 512 (1972).
- (633) Neumann, M., Hertl, W., *J. Chromatogr.*, **65**, 467 (1972).
- (634) Neumann, M., Morales, S., *ibid.*, **74**, 332 (1972).
- (635) Nichikova, P. R., Tember, G. A., Kiselev, N. S., Martynushkina, A. V., Getmanskaya, Z. I., *Neftepererab. Neftekhim.*, **1972**, 34.
- (636) Nigam, R. N., Moolichandra, R., *Anal. Chem.*, **43**, 1683 (1971).
- (637) Nikelly, J. G., *ibid.*, **44**, 623 (1972).
- (638) *ibid.*, p 625.
- (639) *ibid.*, **45**, 1264 (1973).
- (640) Nikolayev, A. M., Vlodayets, M. I., *Zavod. Lab.*, **37**, 769 (1971).
- (641) Noel, D., Meloan, C., *Separ. Sci.*, **7**, 389 (1972).
- (642) Nonaka, A., *Anal. Chem.*, **44**, 271 (1972).
- (643) *ibid.*, **45**, 483 (1973).
- (644) Nonaka, A., *Bunseki Kagaku*, **20**, 416 (1971).
- (645) *ibid.*, p 422.
- (646) Noolchandra, R., Mallik, K. L., *Anal. Chem.*, **44**, 2404 (1972).
- (647) Novak, J., Galbicova-Ruzickova, J., Wicar, S., *J. Chromatogr.*, **60**, 127 (1971).
- (648) Novak, J., Galbicova-Ruzickova, J., Wicar, S., Janak, J., *Anal. Chem.*, **43**, 1996 (1971).
- (649) *ibid.*, **45**, 1365 (1973).
- (650) Novotny, M., Janak, J., *Chem. Listy*, **66**, 693 (1972).
- (651) Novotny, M., Segura, R., Zlatkis, A., *Anal. Chem.*, **44**, 9 (1972).
- (652) Oertel, R. P., Myhre, D. V., *ibid.*, p 1589.
- (653) Okamura, J., *Diss. Abstr.*, **33**, 2509B (1972).
- (654) Okamura, J., Sawyer, D. T., *Anal. Chem.*, **43**, 1730 (1971).
- (655) *ibid.*, **45**, 80 (1973).
- (656) Ono, A., *Nippon Kagaku Zasshi*, **92**, 429 (1971).
- (657) Oppegaard, A., *J. Chromatogr. Sci.*, **10**, 716 (1972).
- (658) Oszczapowicz, J., Golab, J., Pines, H., *J. Chromatogr.*, **64**, 1 (1972).
- (659) Otte, E., *Chromatographia*, **4**, 588 (1971).
- (660) Otte, E., Gurt, J., *ibid.*, **5**, 246 (1972).
- (661) Ottenstein, D. M., *J. Chromatogr. Sci.*, **11**, 136 (1973).
- (662) Ottenstein, D. M., Bartley, D. A., *ibid.*, **9**, 673 (1971).
- (663) Palframan, J., Macnab, J., Crosby, N., *J. Chromatogr.*, **76**, 307 (1973).
- (664) Pan, B. Y. K., *Anal. Instrum.*, **9**, 1 (1971).
- (665) Panina, L., Klinskaya, N., Glazunova, L., Sakadynskii, K., *J. Chromatogr.*, **77**, 51 (1973).
- (666) Papendick, H., *Erdol Kohle*, **24**, 637 (1971).
- (667) Parcher, J. F., Hussey, C. L., *Anal. Chem.*, **45**, 188 (1973).
- (668) Paris, C., Alexandre, P., *J. Chromatogr. Sci.*, **10**, 402 (1972).
- (669) Parr, W., Howard, P., *Anal. Chem.*, **45**, 711 (1973).
- (670) Parr, W., Howard, P., *J. Chromatogr.*, **66**, 141 (1972).
- (671) *ibid.*, **67**, 227 (1972).
- (672) *ibid.*, **71**, 193 (1972).
- (673) Patzelova, V., Volkova, J., *ibid.*, **65**, 255 (1972).
- (674) Paul, G. T., *Chromatographia*, **5**, 192 (1972).
- (675) Pease, E. C., *Anal. Chem.*, **45**, 1584 (1973).
- (676) Pella, E., Colombo, B., *ibid.*, **44**, 1563 (1972).
- (677) Penzias, G. J., *ibid.*, **45**, 890 (1973).
- (678) Persinger, H. E., Shank, J. T., *J. Chromatogr. Sci.*, **11**, 190 (1973).
- (679) Pesek, J. J., Pecsok, R. L., *Anal. Chem.*, **44**, 620 (1972).
- (680) Peterson, R. M., Rodgers, J., *Chromatographia*, **5**, 13 (1972).
- (681) Petrov, A. A., Kolesnikova, L. P., *Zh. Anal. Khim.*, **27**, 1050 (1972).
- (682) Petsev, N. D., *DoKl. Bolgar. Akad. Nauk*, **24**, 1043 (1971).
- (683) Pilgrim, G. W., Keller, R. A., *J. Chromatogr. Sci.*, **11**, 206 (1973).
- (684) Plant, A. F., *Ind. Res.*, **14**(6), 56 (1972).
- (685) Pockington, R., *Anal. Biochem.*, **45**, 403 (1972).
- (686) Pollock, G. E., *Anal. Chem.*, **44**, 634 (1972).
- (687) Popescu, R., Blidisel, I., Grigoriu, D., *Petrol. Gaze (Bucharest)*, **22**, 111 (1971).
- (688) Porcaro, P. J., Shubiak, P., *J. Chromatogr. Sci.*, **9**, 690 (1971).
- (689) Porter, M. L., Burke, J. A., *J. Ass. Offic. Anal. Chem.*, **54**, 1426 (1971).
- (690) Preston Technical Abstracts Co., P. O. Box 312, Niles, Ill. 60648: *Gas Chromatography Literature Abstracts and Index: Journal of Chromatographic Science: and literature retrieval devices and services.*
- (691) Pretorius, V., Hahn, H. H., U.S. Patent **3,594,788** (July 20, 1971).
- (692) *ibid.*, **3,649,498** (Mar. 14, 1972).
- (693) Price, J. G. W., Scott, J. C., Wheeler, L. O., *J. Chromatogr. Sci.*, **9**, 722 (1971).
- (694) Price, S. J. W., Richard, J. P., *Can. J.*

- Chem.*, **50**, 966 (1972).
- (695) Price, S. J. W., Richard, J. P., Rumpfolt, R. C., *ibid.*, **51**, 1397 (1973).
- (696) Purcell, J. E., March, E. W., Adams, R., *Chromatogr. Newslett.*, **1**(1), 14 (1972).
- (697) Purnell, J. H. (to Foxboro Co.), U.S. Patent **3,628,310** (Dec. 21, 1971).
- (698) Purnell, J. H., Ed., "New Developments in Gas Chromatography," Vol. II, Wiley-Interscience, New York, 1973.
- (699) Purnell, J. H., Srivastava, O. P., *Anal. Chem.*, **45**, 1111 (1973).
- (700) Rabinovich, A. S., Sakharov, V. M., *Zh. Anal. Khim.*, **27**, 666 (1972).
- (701) Rabinovich, A. S., Sakharov, V. M., Skop, S. L., *Zavod. Lab.*, **38**, 1299 (1972).
- (702) Radyuk, Z. A., Kabo, G. Y., Andreyevskii, D. N., *Neftekhimiya*, **12**, 679 (1972).
- (703) Rasmussen, R. A., *Amer. Lab.*, **4**(7), 19 (1972).
- (704) *Ibid.*, **5**(2), 99 (1973).
- (705) Rasmussen, K., Rasmussen, S., Svendsen, A. B., *J. Chromatogr.*, **66**, 136 (1972).
- (706) *Ibid.*, **71**, 542 (1972).
- (707) Redshaw, E. S., Hougen, F. W., Baker, R. J., *J. Agr. Food Chem.*, **19**, 1264 (1971).
- (708) Reeve, J., *Diss. Abstr.*, **32**, 5664B (1972).
- (709) Reimendal, R., Sjövall, J., *Anal. Chem.*, **44**, 21 (1972).
- (710) Reiner, E., Hicks, J. J., *Chromatographia*, **5**, 525 (1972).
- (711) Raymond, D., *Flavor Ind.*, **2**, 575 (1971).
- (712) Rezl, V., *J. Chromatogr. Sci.*, **10**, 419 (1972).
- (713) Richard, H. M., Russell, G. F., Jennings, W. G., *ibid.*, **9**, 560 (1971).
- (714) Riedmann, M., "Sixth International Symposium on Chromatography and Electrophoresis," Ann Arbor Sci. Publ., Ann Arbor, 1971, p 277.
- (715) Rijnders, G. W. A., Bleumer, J. P. A., van Krevelen, M., *Usp. Khromatogr.*, **1972**, 65-79.
- (716) Ristau, W. T., Vanderborgh, N. E., *Anal. Chem.*, **44**, 359 (1972).
- (717) Roberts, G. L., Hawkes, S. J., *J. Chromatogr. Sci.*, **11**, 16 (1973).
- (718) Roberts, S. M., *Anal. Chem.*, **44**, 502 (1972).
- (719) Robinson, W. H., Hilton, H. W., *J. Agr. Food Chem.*, **19**, 875 (1971).
- (720) Roethel, D. A. H., Counts, C. R., *Chem. Eng. News*, **49**(47), 90 (1971).
- (721) Roganov, C. N., Kabot, G. Y., Andreyevskii, D. N., *Neftekhimiya*, **12**, 495 (1972).
- (722) Roghinskii, S. Z., Yanovskii, M. I., Berman, A. D., "Aspects in Gas Chromatography," H. G. Struppe, Ed., Akad. Verlag, Berlin, 1971, pp 96-111.
- (723) Roghinskii, S. Z., Yanovskii, M. I., Berman, A. D., "The Fundamentals of Chromatographic Applications in Catalysis," Nauka, Moscow, 1972.
- (724) Rohrschneider, L., *Anal. Chem.*, **45**, 1241 (1973).
- (725) Rohrschneider, L., *J. Chromatogr. Sci.*, **11**, 160 (1973).
- (726) Roper, F. G., *ibid.*, **9**, 697 (1971).
- (727) Rosie, D. M., Barry, E. F., *ibid.*, **11**, 237 (1973).
- (728) Rosmus, J., Deyl, Z., *J. Chromatogr.*, **70**, 221 (1972).
- (729) Ross, R., Shafik, T., *J. Chromatogr. Sci.*, **11**, 46 (1973).
- (730) Rossiter, V., *Chromatographia*, **4**, 354 (1971).
- (731) Rotzsche, H., "Aspects in Gas Chromatography," H. G. Struppe, Ed., Akad.-Verlag, Berlin, 1971, pp 120-9.
- (732) Rozenblit, A. B., Silis, Y. Y., Avots, A. A., Shats, V. D., *Zh. Anal. Khim.*, **28**, 22 (1973).
- (733) Rudenko, B. A., *J. Chromatogr. Sci.*, **10**, 230 (1972).
- (734) Rudenko, B. A., Baidarovtseva, M. A., Kucherov, V. F., *Izv. Akad. Nauk SSSR. Ser. Khim.*, **1972**, 2634.
- (735) Rudnaya, G. V., Pravednikov, A. N., *Lakokrasoch. Mater. Ikh Primen.*, **1972**, (3), 18.
- (736) Rudzinski, W., Suprynowicz, Z., Rayss, J., *J. Chromatogr.*, **66**, 1 (1972).
- (737) Rudzinski, W., Waksmundzki, A., Suprynowicz, A., Rayss, J., *ibid.*, **72**, 221 (1972).
- (738) Rutter, G., Luyten, J., *ibid.*, **74**, 177 (1972).
- (739) Ryba, M., *Chromatographia*, **5**, 23 (1972).
- (740) Saitoh, K., Kambara, T., *Bunseki Kagaku*, **20**, 1375 (1971).
- (741) Sakhorov, V., Beskova, G., Butusova, A., *J. Chromatogr.*, **69**, 71 (1972).
- (742) Sakodinsky, K. I., Klinskaya, N. S., Panina, L. I., *Anal. Chem.*, **45**, 1369 (1973).
- (743) Sakodinsky, K. I., Panina, L. I., *Zh. Anal. Khim.*, **27**, 1024 (1972).
- (744) Samuelsson, B., Hamberg, M., Sweeley, C. C., *Anal. Biochem.*, **38**, 301 (1970).
- (745) Sandoval, A. A., U.S. Patent **3,630,006** (Dec. 28, 1971).
- (746) Sarner, S. F., *J. Chromatogr. Soc.*, **10**, 65 (1972).
- (747) Sato, T., Ohkoshi, S., Takahashi, T., *J. Chromatogr.*, **65**, 413 (1972).
- (748) Scales, J. W., Ed., "Air Quality Instrumentation," Vol. I, Instrument Society of America, Pittsburgh, Pa., 1972.
- (749) Schaefer, B. A., *J. Chromatogr. Soc.*, **10**, 110 (1972).
- (750) Schaefer, B. A., Douglas, D. M., *ibid.*, **9**, 612 (1971).
- (751) Schay, G., *Usp. Khromatogr.*, **1972**, 179-92.
- (752) Scheller, W. A., Petricek, J. L., Young, G. C., *Ind. Eng. Chem., Fundam.*, **11**, 53 (1972).
- (753) Schep, R. A., De Clerk, K., *J. Chromatogr. Sci.*, **10**, 530 (1972).
- (754) Schieterh, G. A., Greiner, K., *Chromatographia*, **5**, 70 (1972).
- (755) Schnell, E., Fuchs, H., *Mikrochim. Acta*, **1972**, 97.
- (756) Schoengold, D., *Diss. Abstr.*, **32**, 6261B (1972).
- (757) Schoengold, D. M., Stewart, W. H., *Anal. Chem.*, **44**, 884 (1972).
- (758) Schomburg, G., Dielmann, G., *J. Chromatogr. Sci.*, **11**, 151 (1973).
- (759) Schomburg, G., Henneberg, D., "Aspects in Gas Chromatography," H. G. Struppe, Ed., Akad. Verlag, Berlin, 1971, pp 130-40.
- (760) Schomburg, G., Weeke, F., Weimann, B., Ziegler, E., *J. Chromatogr. Sci.*, **9**, 735 (1971).
- (761) Schomburg, G., Ziegler, E., *Chromatographia*, **5**, 96 (1972).
- (762) Schulze, P., Kaiser, K. H., *ibid.*, **4**, 381 (1971).
- (763) Schultz, T. H., Flath, R. A., Mon, T. R., *J. Agr. Food Chem.*, **19**, 1060 (1971).
- (764) Schurig, V., Bear, J. L., Zlatkis, A., *Chromatographia*, **5**, 301 (1972).
- (765) Schutte, L., Koenders, E., *J. Chromatogr.*, **76**, 13 (1973).
- (766) Schuy, K. D., Hunneman, D. H., *Chem.-Ztg.*, **95**, 633 (1971).
- (767) Schwartz, R. D., Matthews, R. G., Irvine, D. M., Durbin, D. E., Fruge, J. M., *Anal. Chem.*, **45**, 1280 (1973).
- (768) Schwartz, R. D., Matthews, R. G., Rountree, J. E., Irvine, D. M., *ibid.*, **43**, 2000 (1971).
- (769) Schwedt, G., Ruessel, H. A., *Chromatographia*, **5**, 242 (1972).
- (770) Scolnick, M. E., U.S. Patent **3,589,869** (June 29, 1971).
- (771) Seiber, J., Markie, J., *Bull. Environ. Contam. Toxicol.*, **7**(2/3), 72 (1972).
- (772) Selucky, M. L., *Chromatographia*, **4**, 425 (1971).
- (773) *Ibid.*, **5**, 359 (1972).
- (774) Semenchenko, L. V., *Zh. Anal. Khim.*, **27**, 1602 (1972).
- (775) Serpinet, J., *J. Chromatogr.*, **68**, 9 (1972).
- (776) *Ibid.*, **77**, 289 (1973).
- (777) Serpinet, J., Robin, J., *C. R. Acad. Sci.*, **272**, 1765 (1971).
- (778) Sevcik, J., *Chromatographia*, **4**, 195 (1971).
- (779) Shaler, R., Padden, J., *J. Pharm. Sci.*, **61**, 1851 (1972).
- (780) Shaler, R. C., Prichard, W. W., *J. Ass. Offic. Anal. Chem.*, **55**, 832 (1972).
- (781) Shapunov, L. A., Parniuk, N. I., Gosteminskaya, T. V., Kuratova, T. S., *Zavod. Lab.*, **37**, 1066 (1971).
- (782) Shcherbina, T. M., Bondarev, V. B., Sokolov, A. V., Vitt, S. V., *Izv. Akad. Nauk SSSR, Ser. Khim.*, **1972**, 443.
- (783) Shifman, V. S., Zhestkov, D. K., Galpern, G. D., *Zavod. Lab.*, **38**, 540 (1972).
- (784) Shimoishi, Y., *Bull. Chem. Soc. Japan*, **44**, 3370 (1971).
- (785) Shlapkova, Y. P., Niemkovich, V. A., *Tepl. Massoperenos App. Dispers. Sist. Minsk*, **1970**, 76-82.
- (786) Shlyakhov, A. F., Latukhova, A. G., *Zavod. Lab.*, **38**, 160 (1972).
- (787) Shlyakhova, I. V., Zelskaya, L. I., *Khim. Prom.*, **48**, 67 (1972).
- (788) Shuchowizkij, A. A., Sazonov, M. L., Gelman, L. G., Schwarzman, W. P., *Chromatographia*, **4**, 547 (1971).
- (789) Sidorov, R. I., Batyrev, Y. A., *Zh. Anal. Khim.*, **27**, 1428 (1972).
- (790) Sidorov, R. I., Denisenko, A. N., Ivanova, M. P., Reznikov, S. A., Khovostikova, A. A., Borovskaya, I. S., *ibid.*, p 1013.
- (791) Sieck, R., *Diss. Abstr.*, **32**, 1412B (1971).
- (792) Sieck, R., Banks, C. V., *Anal. Chem.*, **44**, 2307 (1972).
- (793) Silayeva, I. A., Zhukhovitskii, A. A., Sazonov, M. L., Shvartsman, V. P., *Zavod. Lab.*, **38**, 1297 (1972).
- (794) Simmonds, P. G., Shoemake, G. R., Lovelock, J. E., Lord, H. C., *Anal. Chem.*, **44**, 860 (1972).
- (795) Simon, A., Otvos, I., Palyi, G., *Kem. Közlem.*, **37**, 81 (1972).
- (796) Sine, H. E., McKenna, M. J., Law, M. R., Murray, M. H., *J. Chromatogr. Sci.*, **10**, 297 (1972).
- (797) Sivtsova, E. V., *Zh. Prikl. Khim.*, **45**, 201 (1972).
- (798) *Ibid.*, p 210.
- (799) Smith, D. H., Olsen, R. W., Walls, F. C., Burlingame, A. L., *Anal. Chem.*, **43**, 1796 (1971).
- (800) Smith, E. D., *J. Chromatogr. Sci.*, **10**, 34 (1972).
- (801) Smith, R., Leong, A., *J. Pharm. Sci.*, **61**, 316 (1972).
- (802) Smuts, T. W., Pretorius, V., *Separ. Sci.*, **6**, 583 (1971).
- (803) Sniegowski, P. J., *J. Chromatogr. Sci.*, **10**, 644 (1972).
- (804) Snyder, R. E., *ibid.*, **9**, 638 (1971).
- (805) Snyder, R. E., *ibid.*, **10**, 487 (1972).
- (806) Soda, R., *Bull. Chem. Soc. Japan*, **44**, 3032 (1971).
- (807) Sojak, L., Hrivnak, J., Krupcik, J., Janak, J., *Anal. Chem.*, **44**, 1701 (1972).
- (808) Sojak, L., Hrivnak, J., Majer, P., Janak, J., *ibid.*, **45**, 293 (1973).
- (809) Sojak, L., Krupcik, J., Tesarik, K., Janak, J., *J. Chromatogr.*, **65**, 93 (1972).
- (810) Sojak, L., Majer, P., Skalac, P., Janak, J., *ibid.*, **65**, 137 (1972).
- (811) Sokolov, D. N., Vakin, N. A., *J. Chromatogr. Sci.*, **10**, 417 (1972).
- (812) Sokolov, D. N., Vakin, N. A., USSR Patent **268,001** (July 31, 1970).
- (813) Sokolov, D. N., Vakin, N. A., *Zavod. Lab.*, **36**, 513 (1970).
- (814) Soloviev, Y. V., *ibid.*, **38**, 1405 (1972).
- (815) Sowinski, E. J., Suffet, I. H., *Anal. Chem.*, **44**, 2237 (1972).
- (816) Sowinski, E. J., Suffet, I. H., *J. Chromatogr. Sci.*, **9**, 632 (1971).
- (817) Spangler, W. J., Spigarelli, J. L., Rose, J. M., Miller, H. M., *Science (London)*, **180**, 192 (1973).
- (818) Sperling, A., *J. Chromatogr. Sci.*, **10**, 268 (1972).
- (819) Stafford, D., *Diss. Abstr.*, **33**, 1971B (1972).
- (820) Staszewski, R., *Tluszczze Jadaine*, **16**, 165 (1972).
- (821) Stewart, W. H., *Anal. Chem.*, **44**, 1547 (1972).
- (822) Stiepnienko, V. Y., Krichmar, S. I., *Zavod. Lab.*, **38**, 1200 (1972).
- (823) Struble, D. L., *J. Chromatogr. Sci.*, **10**, 57 (1972).
- (824) Struppe, H. G., Ed., "Aspects in Gas Chromatography," Akad.-Verlag, Berlin, 1971; English translation available from Preston Technical Abstr. Co., P.O. Box 312, Niles, Ill. 60648.
- (825) Styskin, Y. L., Taradai, Y. P., Sharpanova, I. K., *Zavod. Lab.*, **37**, 1182 (1971).
- (826) Suprynowicz, Z., *Chem. Anal. (Warsaw)*, **18**, 15 (1973).

- (827) Suprynowicz, Z., Waksmundzki, A., Rudzinski, W., *J. Chromatogr.*, **67**, 21 (1972).
- (828) *Ibid.*, **72**, 5 (1972).
- (829) Sussman, M. V., Astill, K. N., Rombach, R., Cerullo, A., Chen, S. S., *Ind. Eng. Chem., Fundam.*, **11**, 181 (1972).
- (830) Sutre, P., Malenge, J. P., *Chromatographia*, **5**, 141 (1972).
- (831) Sutter, F., Fuchs, P., *Arzneim.-Forsch.*, **22**, 1553 (1972).
- (832) Suzuki, M., Smith, J. M., *Chem. Eng. Sci.*, **26**, 221 (1971).
- (833) Svoboda, P., Vaisarova, V., Chcalcovsky, V., *Collect. Czech. Chem. Commun.*, **37**, 2258 (1972).
- (834) Swann, M. H., Adams, M. L., Esposito, G. G., *Anal. Chem.*, **45**, 39R (1973).
- (835) Swansiger, J. T., Dickson, F. E., *ibid.*, p 811.
- (836) Swingle, R., *Diss. Abstr.*, **32**, 3225B (1971).
- (837) Swingle, R. S., Rogers, L. B., *Anal. Chem.*, **44**, 1415 (1972).
- (838) Sybilska, D., Malinowska, K., Siekierska, M., Bylina, J., *Chem. Anal. (Warsaw)*, **17**, 1031 (1972).
- (839) Sytienko, T. P., *Tr. Groznensk. Neft. Inst. Sb.*, **33**, 142 (1971).
- (840) Szentirmai, Z., Tarjan, G., Takacs, J., *J. Chromatogr.*, **73**, 11 (1972).
- (841) Takacs, J., Kralik, D., Francioso, P., *Magy. Kem. Foly.*, **78**, 43 (1972).
- (842) Takacs, J., Nyireadi, S., Fischer, G., Tarjan, G., Szita, K., *ibid.*, p 403.
- (843) Takacs, J., Szentirmai, Z., Molnar, E., Kralik, D., *J. Chromatogr.*, **65**, 121 (1972).
- (844) Takacs, J., Talas, Z., Bernath, I., Czako, G., Fischer, A., *ibid.*, **67**, 203 (1972).
- (845) Takahashi, Y., Moore, R. T., Joyce, R. J., *Amer. Lab.*, **4**(7), 31 (1972).
- (846) Takemura, I., *Bunseki Kagaku*, **22**, 36 (1973).
- (847) Taramasso, M., Sakodynskii, K. I., *Usp. Khromatogr.*, **1972**, 248.
- (848) Tassios, D. P., *Int. Eng. Chem., Process Des. Develop.*, **11**, 43 (1972).
- (849) Taylor, P., Culp, R., Lochmuller, C., Rogers, L. B., *Separ. Sci.*, **6**, 841 (1971).
- (850) Tesarik, K., Necasova, M., *J. Chromatogr.*, **65**, 39 (1972).
- (851) *Ibid.*, **75**, 1 (1973).
- (852) Tham, M. K., Gubbins, K. E., *J. Chem. Soc., Faraday Trans. 1*, **1972**, 1339.
- (853) Thompson, R. H., Jr., Pearson, A. M., Banks, K. A., *J. Agr. Food Chem.*, **20**, 185 (1972).
- (854) Thornburg, W., *Anal. Chem.*, **45**, 151R (1973).
- (855) Thurman, R., *Diss. Abstr.*, **32**, 127B (1971).
- (856) Thurman, R., Burke, M., *J. Chromatogr. Sci.*, **9**, 181 (1971).
- (857) Tijssen, R., Wittenbrood, R. T., *Chromatographia*, **5**, 286 (1972).
- (858) Tomazic, B. B., O'Laughlin, J. W., *Anal. Chem.*, **45**, 1519 (1973).
- (859) Trash, C. R., *J. Chromatogr. Sci.*, **11**, 196 (1973).
- (860) Trubin, A. M., USSR Patent **269,572** (Aug. 4, 1970).
- (861) Tsitsishvili, G. V., Andronikashvili, T. G., *Usp. Khromatogr.*, **1972**, 226-35.
- (862) Tsuda, T., Ojika, Y., Izuda, M., Fujishima, I., Ishii, D., *J. Chromatogr.*, **69**, 194 (1972).
- (863) Tunistra, L. G. M. T., de Graaff, J. B. H. D., *Chromatographia*, **4**, 468 (1971).
- (864) Uhdeova, J., Janak, J., *J. Chromatogr.*, **65**, 179 (1972).
- (865) Umanskaya, A. A., Makarenkova, R. M., Sokolov, N. M., Zhavoronkov, N. M., *Zh. Anal. Khim.*, **25**, 1211 (1970).
- (866) Umiilin, V. A., Tyutyayev, I. N., *Izv. Akad. Nauk SSSR. Ser. Khim.*, **1972**, 1262.
- (867) Umiilin, V. A., Vanchagova, V. K., *ibid.*, p 651.
- (868) Underhill, D. W., *Anal. Chem.*, **45**, 1258 (1973).
- (869) Ustinovskaya, I. A., Gavrilina, L. Y., Chirkova, V. M., Malakhov, V. V., *Khim. Prom.*, **48**, 394 (1972).
- (870) Uthe, J. F., Solomon, J., Grift, B., *J. Ass. Offic. Anal. Chem.*, **55**, 583 (1972).
- (871) Utsunomiya, K., Shigematsu, T., *Anal. Chim. Acta*, **58**, 411 (1972).
- (872) Utsunomiya, K., Shigematsu, T., *Bull. Chem. Soc. Japan*, **45**, 303 (1972).
- (873) Van Dalen, J. P. J., *Chromatographia*, **5**, 354 (1972).
- (874) Van den Berg, P. M. J., Cox, T. P., *ibid.*, p 301.
- (875) Van de Wiel, H., Tommassen, P., *J. Chromatogr.*, **71**, 1 (1972).
- (876) van Rensburg, J. F. J., Mouton, P. L., Pretorius, V., *J. Chromatogr. Sci.*, **10**, 580 (1972).
- (877) Varsano, J., Gilbert, S., *J. Pharm. Sci.*, **62**, 87, 92 (1973).
- (878) Vatulya, N. M., Sukhorukov, O. A., *Zavod. Lab.*, **38**, 133 (1972).
- (879) Vatulya, N. M., Sukhorukov, O. A., Ventzel, M. D., *Zh. Fiz. Khim.*, **45**, 2633 (1971).
- (880) Venkataraghavan, R., McLafferty, F. W., *Chem. Technol.*, **1972**, 364.
- (881) Verdin, D., *Anal. Chem.*, **43**, 1909 (1971).
- (882) Vermont, J., Guillemin, C. L., *ibid.*, **45**, 775 (1973).
- (883) Vernon, F., *J. Chromatogr.*, **60**, 406 (1971).
- (884) Versino, B., Rissi, G., *Chromatographia*, **4**, 331 (1971).
- (885) Verzele, M., Verstappe, M., Sandra, P., Van Luchene, E., Vuye, A., *J. Chromatogr. Sci.*, **10**, 668 (1972).
- (886) Vidal-Madjar, C., Guiochon, G., "Aspects in Gas Chromatography," H. G. Struppe, Ed., *Akad.-Verlag, Berlin*, 1971, pp 191-205.
- (887) Vidal-Madjar, C., Guiochon, G., *J. Chromatogr. Sci.*, **9**, 664 (1971).
- (888) Vignalok, R. V., Vigdergauz, M. S., *Izv. Akad. Nauk SSSR. Ser. Khim.*, **1972**, 715.
- (889) Vigdergauz, M. S., *Zh. Anal. Khim.*, **27**, 980 (1972).
- (890) Vigdergauz, M. S., Pomazanov, V. V., Bogdanchikov, A. I., Gunchenko, E. I., *Izv. Akad. Nauk SSSR. Ser. Khim.*, **1972**, 646.
- (891) Vigdergauz, M. S., Pomazanov, V. V., *ibid.*, **1971**, 2835.
- (892) Vigdergauz, M. S., Semkin, V. I., *Usp. Khim.*, **40**, 1073 (1971).
- (893) Vigdergauz, M. S., Vignalok, R. V., *Neftekhimiya*, **11**, 141 (1971).
- (894) Vitenburg, A. G., Pospelova, A. K., Ioff, B. V., *ibid.*, **12**, 623 (1972).
- (895) Voykevich, S. A., Shchedrina, M. M., Solovieva, N. P., Rudolff, T. A., *Maslob.-Zhir. Prom.*, **37**, 27 (1971).
- (896) Wade, R. L., Cram, S. P., *Anal. Chem.*, **44**, 131 (1972).
- (897) Wade, R. L., Cram, S. P., *J. Chromatogr. Sci.*, **10**, 622 (1972).
- (898) Wadelin, C. W., Morris, M. C., *Anal. Chem.*, **45**, 333R (1973).
- (899) Waksmundzki, A., Leboda, R., Suprynowicz, Z., *Chem. Anal. (Warsaw)*, **17**, 1233 (1972).
- (900) Waksmundzki, A., Rudzinski, W., Suprynowicz, Z., Rayss, J., *J. Chromatogr.*, **74**, 3 (1972).
- (901) Waksmundzki, A., Suprynowicz, Z., Leboda, R., *ibid.*, **61**, 217 (1971).
- (902) Waksmundzki, A., Suprynowicz, Z., Leboda, R., Rayss, J., Kusak, R., *Chem. Anal. (Warsaw)*, **16**, 43 (1971).
- (903) Walker, J. Q., *Chromatographia*, **5**, 547 (1972).
- (904) Walker, J. Q., Jackson, M. T., Jr., Maynard, J. B., "Chromatographic Systems; Maintenance and Troubleshooting," Academic Press, New York, 1972.
- (905) Wallace, D. L., *Amer. Lab.*, **4**(9), 67 (1972).
- (906) Warden, T., Schoenig, D., *J. Chromatogr. Sci.*, **10**, 727 (1972).
- (907) Wardsenik, W., Staszewski, R., *Pluszcze Jadatne*, **15**, 289 (1971).
- (908) Watson, G., Williams, J., *J. Chromatogr.*, **67**, 221 (1972).
- (909) Weems, G. W., Hoffman, C. A., Howard, A. R., *J. Chromatogr. Sci.*, **9**, 444 (1971).
- (910) Wehling, I., *Chromatographia*, **5**, 197 (1972).
- (911) Weiner, P., Dack, C., Howery, D., *J. Chromatogr.*, **69**, 249 (1972).
- (912) Weiner, P. H., Parger, J. F., *J. Chromatogr. Sci.*, **10**, 612 (1972).
- (913) Weinlaender, W., Hoehlein, G., *Chromatographia*, **5**, 258 (1972).
- (914) Weland, J. M., Muir, A. R., *ibid.*, p 136.
- (915) Wentworth, W. E., "Recent Advances in Gas Chromatography," I. I. Domsy and J. A. Perry, Ed., Marcel Dekker, New York, 1971, pp 105-24.
- (916) Wernliand, R. F., U.S. Patent **3,626,181** (Dec. 7, 1971).
- (917) West, C. D., *Anal. Chem.*, **42**, 811 (1970).
- (918) Wheals, B. B., Noble, W., *Chromatographia*, **5**, 553 (1972).
- (919) Wicar, S., Novak, J., J. Ruseva-Rakshieva, N., *Anal. Chem.*, **43**, 1945 (1971).
- (920) Wicarova, O., Novak, J., Janak, J., *J. Chromatogr.*, **65**, 241 (1972).
- (921) Williams, W. G., Carritt, D. E., *Anal. Chem.*, **44**, 2119 (1972).
- (922) Wise, P. M., Hanson, R. H., *ibid.*, p 2393.
- (923) Wittenbrook, R. T., *Chromatographia*, **5**, 454 (1972).
- (924) Wolf, E., Smith, J. M., *Ind. Eng. Chem. Fundam.*, **11**, 413 (1972).
- (925) Wolf, C. J. (to McDonnell Douglas Corp.), U.S. Patent **3,607,075** (Sept. 21, 1972).
- (926) Wolf, W. R., Hughes, B. M., Tiernan, T. O., Sievers, R. E., Taylor, M. L., *Anal. Chem.*, **44**, 616 (1972).
- (927) Wright, H. W., Shu-Shin Wang, W., *NASA Tech. Brief* **71-10474**, 1971.
- (928) Wurst, M., Churacek, J., *Collect. Czech. Chem. Commun.*, **36**, 3497 (1971).
- (929) Wurst, M., Churacek, J., *J. Chromatogr.*, **70**, 1 (1972).
- (930) Yalkowsky, S., Flynn, G., Slunick, T., *J. Pharm. Sci.*, **61**, 852 (1972).
- (931) Yanovskii, M. I., Berman, A. D., *Usp. Khim.*, **42**, 147 (1973).
- (932) Yanovskii, S. M., Alksnis, O. N., *Zh. Anal. Khim.*, **27**, 1834 (1972).
- (933) Yanovskii, S. M., Silayeva, I. A., Gelman, L. G., Shvartsman, V. P., Sazonov, M. L., *Zavod. Lab.*, **38**, 526 (1972).
- (934) Yersanian, J. A., Sloman, K. G., Foltz, A. K., *Anal. Chem.*, **45**, 77R (1973).
- (935) Yermakov, V. V., "Gas Chromatographic Methods for Pesticide Determination in Biological Objects," Nauka, Moscow, 1972.
- (936) Yevdokimova, S. P., Isakova, N. A., Yevdokimov, V. F., *Zh. Anal. Khim.*, **26**, 806 (1971).
- (937) Yinon, J., Boettger, H., *Chem. Instrum.*, **4**, 103 (1972).
- (938) Young, C., *J. Chem. Soc., Faraday Trans. 2*, **1972**, 580.
- (939) Yudina, I., Volkov, S., Zel'vinskii, V., Sakodynskii, K., *J. Chromatogr.*, **77**, 41 (1973).
- (940) Yusfin, V. S., Okhotnikov, B. P., Rotin, V. A., Bondarenko, I. V., *Zavod. Lab.*, **38**, 529 (1972).
- (941) Zado, F. M., Juvet, R. S., in "Aspects in Gas Chromatography," H. G. Struppe, Ed., *Akad.-Verlag, Berlin*, 1971, pp 206-11.
- (942) Zel'vinskii, V. Y., Volkov, S. A., Sakodynskii, K. I., *Chromatographia*, **5**, 217 (1972).
- (943) Zenz, H., Klaushofer, H., Szep, H., *ibid.*, p 343.
- (944) Zerba, E., Ruveda, M., *J. Chromatogr.*, **68**, 245 (1972).
- (945) Zershchikova, V. P., Dorogochinskii, A. Z., Pecheritsa, S. A., Balashova, V. V., *Tr. Groznensk. Neft. Inst.*, **33**, 121 (1971).
- (946) Zhukov, N. F., Lipavskii, V. N., Markelov, V. F., Sokolin, G. F., USSR Patent **277,386** (Oct. 29, 1970).
- (947) Zhukhovitski, A. A., *Zh. Anal. Khim.*, **27**, 971 (1972).
- (948) Zhukhovitski, A. A., Sazonov, M. L., Gelman, L. G., Shvartsman, V. P., *Zh. Fiz. Khim.*, **45**, 655 (1971).
- (949) Ziegler, E., Henneberg, D., Schomburg, G., *Anal. Chem.*, **42**, (9), 51A (1970).
- (950) Zilahy, K. I., Hollosy, K., Takacs, J., *Olaj Szappan Kozmetika*, **21**(2), 52 (1972); *ibid.*, **21**(3), 82 (1972).
- (951) Zlatkis, A., Ed., "Advances in Chromatography, 1973," University of Houston Publishers, Houston, Texas, 1973.
- (952) Zlatkis, A., Fenimore, D., *Usp. Khromatogr.*, **1972**, 236-47.
- (953) Zoccolillo, L., Liberti, A., *J. Chromatogr.*, **77**, 69 (1973).
- (954) Zorin, A. D., Kuznietsova, T. S., *Zh. Anal. Khim.*, **27**, 1434 (1972).
- (955) Zweig, G., Sherma, J., "Gas Chromatographic Analysis," Vol. VI of "Analytical Methods for Pesticides and Plant Growth Regulators," G. Zweig, Ed., Academic Press, New York, 1972.