

cationic or anionic polystyrene, polyacrylic, polymethacrylic, or phenolic matrix-forming exchange materials, with acidic or basic groups such as sulfonic, phosphoric, phosphonic, or carboxylic acids and amino-, trimethyl ammonium, dimethyl ethanolamine, or quaternary ammonium groups. Drobot et al. (43K) investigated the sulfur, nitrogen, and oxygen compounds in a polar fraction isolated by silica gel chromatography from petroleum distillates boiling above 300 °C; the polar fractions were separated into benzene-soluble and benzene-alcohol-soluble portions; the benzene-soluble portion contained large amounts of high-molecular-weight alcohols and carbonyl compounds, while the benzene-alcohol portion was rich in carboxylic acids, sulfoxides, quinones, and aromatic hydrocarbons; carbazoles, indoles, and pyrroles were present in both fractions.

Mukhopadhyay and Mukhopadhyay (115K) reviewed the literature for the nature, occurrence, and adverse effects on refinery processes of nonhydrocarbon constituents in petroleum, including sulfur, oxygen, nitrogen, and organometallic compounds; the processes for their effective removal are surveyed, and representative constituents are listed with their general formulas and occurrences in crude oil, straight-run products, and cracked products.

Chlorine. The determination of chlorine in petroleum or petrochemicals was the subject of ten papers. Fernandez et al. (46K) determined traces of chlorine in naphthas by vaporizing a sample in a stream of carbon dioxide and burning it in an air-oxygen-carbon dioxide atmosphere; the combustion products were absorbed in hydrogen peroxide, and the chloride was determined by the addition of aqueous ferric nitrate/mercuric thiocyanate reagent and measurement of the extinction at 460 nm; bromine and iodine interfere. Franks and Pullen (47K) describe a technique for the determination of trace amounts of chlorine by liquid chromatography with a potentiometric sensor; a micro metering pump circulates a mobile phase through a narrow-bore tube containing ion-exchange resin in the metal ion form; the sample is injected into the system and the separated chloride is detected with a pair of silver-silver chloride electrodes, one in the flowing stream and one as reference. A three-column gas chromatographic method for analysis of chlorine-rich gaseous effluents was developed by Amouroux and Foll (7K); the columns involved were Chromosorb T for hydrogen chloride, chlorine, and acetylene; silica gel for hydrogen, methane, ethylene, and acetylene; and Chromosorb T with 7% Apiezon L for chlorinated hydrocarbons. Two papers (10K, 62K) concerning the determination of chlorine and sulfur are discussed briefly in the "Sulfur" section of this review.

Ramakrishnan and Subramanian (131K) analyzed mixtures of toluene and its side-chain-chlorinated derivatives by chromatography on Celite 545 coated with 15% DC 200. Vinyl chloride in industrial atmospheres can be monitored by absorption on activated charcoal followed by thermal desorption of the monomer directly onto an analytical chromatographic column according to Ahlstrom et al. (4K). Levine et al. (93K) developed a method to measure worker exposure to vinyl chloride monomer which overcomes the difficulties of the method proposed by OSHA; gases are sampled and stored in aluminized three-layer sample bags, and the contents of the bags are analyzed directly by gas chromatography on a column of Carbowax 400 on Porasil. Hofstader et al. (64K) showed that naturally occurring sulfur compounds of moderate electron affinity interfered with the electron capture gas chromatographic analysis for polychlorinated biphenyls in petroleum products.

Sapiro et al. (138K) reported the nitrogen and chloride content and the amount of hydrogen chloride evolved during distillation at 200 to 300° for five desalinated Russian petroleums.

Analytical and Process Instrumentation

J. W. Loveland and C. N. White

Suntech, Inc., Newtown Square, Pa.

In this year's review we have retained the same section

headings as were used in 1975. Because of the frequent reference to familiar techniques and terminology we are again using abbreviations to aid the reader in his perusal of the text.

Abbreviations recommended by *Chemical Abstracts* will be used and are not itemized here. Other abbreviations are as follows: AA, atomic absorption; IR, infrared; UV, ultraviolet; MS, mass spectrometry; GC, LC, TLC, gas, liquid, and thin-layer chromatography, respectively; XRA, XRD, XRF, X-ray absorption, diffraction, and fluorescence, respectively; NMR, nuclear magnetic resonance; TC, thermal conductivity (detector); FID, flame ionization detector; PFD, flame photometric detector; std dev, standard deviation; vis., viscosity; psig, pounds per square inch gauge; o.d., i.d., outer and inner diameter; in., inch; and HC, hydrocarbon(s).

In preparing this review, it was evident that there was considerable activity in several areas. In the laboratory, GC continued to be the most widely used technique, and LC showed increased usage over the 1975 review. The most active areas of work were Specific Compounds and Type Compounds, and Improved Instrumentation, as in the previous review, while Elemental Analysis and Pollution Analysis and Instrumentation showed considerably less activity. In the process instrumentation area there was little activity in Elemental Analyzers, except sulfur, a greater interest in Pollution systems and Physical Property systems when level, temperature, and pressure instruments are included with the analyzers, and a large increase in activity in Improved Instrumentation, particularly with computer and control devices.

Whereas previous reviews covered review papers in the introduction, this year we will include only those of broad scope. Other review articles of a specific nature will be included in the appropriate sections.

The following are recommended for a general reading and cover a broad spectrum of techniques and/or applications.

Broad Reviews. Kroll (130L) gave a short overview of measurement analysis problems related to petroleum products and environmental air and water sampling and testing. He discussed the nature of the total measurement process, the role of the chemist, statistician, computer, and the ASTM in reaching decisions on quality assurance.

Shved and Kuzevanova (205L) discussed the use of chromatography, spectroscopy, and potentiometry in quality control laboratories in the manufacture of by-product coke. Machida (152L) reviewed the laboratory use of GC, UV, and visible spectroscopy and AA in petrochemical plants.

Kerenyi et al. (119L) reviewed methods for determining the composition of gasoline, lubricating oils, refined petroleum products, vacuum residues, bitumens, and petrochemical products.

In the process instrumentation area, Clevett (45L) has written a book with 470 pages on process stream analysis. Several articles have reviewed the state of the art in the broad areas of analyzer measurement and automation in the processing industry. Becker (18L) covers the scope of control technology for environmental protection to space probes; the purpose and structure of control systems; new measuring systems, such as fluid temperature sensors, and new developments in process computers. Simeone (207L) reviewed the function performed by some 60 process analyzers at an oil refinery. The chemical laboratory has the prime responsibility for purchase, installation, operation, and maintenance of the equipment and regularly checks analyzer results vs. lab tests. Statistical analysis to establish reliability of one analyzer is given. Kikuchi (121L) presents a survey dealing with the maintenance of on-line analyzers for high accuracy. The report includes equipment, personnel, inspection methods, and record keeping methods. Foster (73L) discusses the advantages, limitations, and applications of typical instruments and analyzers and provides guidelines for selecting on-line process analyzers. The list of instruments and analyzers discussed is too lengthy to give here but includes all of those commonly used in the petroleum industry. Foster also covers sampling and prepackaged analyzer systems. Schiele (196L) reviews the measurement of: liquid level in distillation columns, flow of fuel oil and gas, viscosity, temperature in chemical reactions and flames, composition of waste gases, water and atmosphere by GC and spectrometric methods, and electrical parameters by analog and digital techniques (82 refs.). At an NPRA meeting (107L) eighteen questions and answers on analyzers

and instruments were presented verbatim and covered analyzers for S, H₂S, H₂, H₂O, NH₃, O₂ for a variety of refining problems. Other items covered were sampling and maintenance problems. Zakaib (241L) reviewed analytical methods, techniques, and instrumentation for process studies and control. The survey covers the use of on-line GC and spectroscopic analysis to optimize conversion, catalyst life, and product distribution in the refinery; GC's for distillation control, MS and GC data for modeling and new control methods based on analysis of polycyclic aromatic coke precursors, control systems for alkylation, and Claus sulfur units. Yamashita (239L) reviews analyzers for high-sensitivity, high-speed, simultaneous multicomponent and maintenance free analyses in processing industries. Pal (175L) covers the present status and probable future trend of process instrumentation, centralized control, and pneumatic vs. electronic systems. Swaroop (215L) surveys the development and trends in process instruments as plants become larger and more complex. Covered are computers, electronic vs. pneumatic instrumentation, trends in sensors for temperature, strain, flow, and pressure, analyzers, control hardware, DDC and feedforward analog controllers. The systems engineering approach and growth of the Indian process instrumentation industry is discussed. Whelan (235L) discusses instrumentation costs in the hydrocarbon processing industry. Graphs are developed for checking costs in the early stages of a project. Factors considered are instrumentation types, labor and material and installation costs, checkout and testing, freight and packaging, control panel revisions, computer hardware and software, analytical systems and environmental regulations on noise control.

ELEMENTAL ANALYSIS

Laboratory Analyses. Thomerson and Thompson (222L) reviewed the advantages and disadvantages of flame and nonflame atomization techniques and listed typical detection limits. Vapor techniques for Hg and elements that form volatile hydrides were outlined and the various electrothermal atomization devices were described briefly, with reference to the following applications: clinical, metallurgical, petroleum, foodstuffs, chemicals, paper, air, water, and effluents. *Process Engineering (London)* (184L) carried an article describing the use of a Hilger and Watts AA photometer to measure Pb and other metals in used and re-refined oils with an analysis time in minutes. Samples in carrier liquids are sprayed into the flame and the decrease in intensity of transmitted light is detected.

A nondispersive XRF analyzer was described by Tittarelli (224L) for the rapid determination of S (0.08 to 4%) in petroleum products. The instrument can be adapted for the determination of Pb in commercial gasolines. Oshima et al. (172L) described two data processing systems for qualitative analysis by XRD and quantitative analysis by XRF for identifying CaSO₄ and determining P in asphalt.

Newton et al. (166L) described the determination of trace noble gases in air and natural gas. The gas samples were pre-concentrated by reaction with Ca at 900–1000° and analyzed by automated MS. Methods of relating the concentrated sample to the original sample, and the preparation of gas standards are given. Accuracy at 25 ppm was determined and precision discussed.

Containers for weighing liquid samples in the O₂ flask combustion method in organic microanalysis were described by Nara and Hasegawa (165L). Commercial cellulose tape was used to make the containers, and the size and shape suitable for microanalysis were studied. The roll-type container minimized sample vaporization better than the bag type, and the size of the attached filter paper had a marked effect on results.

Process Analyzers. The determination of total sulfur was the main emphasis of on-line elemental analyzers the past few years. Trost (226L) used a combination of high and low γ -rays to determine the density and S content, respectively, while compensating for the C to H ratio of the sample by continuous measurement of the H content with a neutron probe using an Am-Be source. An accuracy of $\pm 0.01\%$ for S and $\pm 1 \text{ g/dm}^3$ for density can be achieved for various type hydrocarbons. Gamage and Topham (80L) describe the development of an on-line nondispersive XRF unit to determine S in distillates and residual fuels in the range of 0 to 4 wt %. The precision

(95% confidence limit) is 0.03 for distillates and 0.05% for residuals. Williams and Haggard (238L) designed a special flow cell for on-line XRF analysis of S in hydrocarbon oils. The cell has a single radiation window of polyimide film supported by a metal grid and its volume can be varied by means of removable solid inserts.

Fasching and Patton (69L) described a pulse-counting system that improves the energy resolution of less-precise γ radiation detectors, e.g., NaI(Tl) by minimizing pulse-pile-up distortion. A pile-up detector selects either a fast or slow processing channel, depending on whether more than one pulse is recorded during the resolving time of the system. This system has been used for the determination of S in a continuous flow of coal.

SPECIFIC COMPOUNDS AND TYPE COMPOUNDS

Laboratory GC Methods. Deans (58L) discussed the laboratory GC in the context of the total analysis requirements for process control of a petrochemical plant. Berezkin and Nametkin (23L) reviewed recent literature on improved separation methods, combinations of chromatography with physical or chemical methods, accelerated chromatographic analysis and GC methods and apparatus for the analysis of pollutants in air and water. Applications of gel permeation chromatography in petrochemistry were reviewed by Hashimoto and Kato (92L). The following topics were covered: instruments, commercially available column packing, characterization of large molecules, methods for oligomer separation, identification and fingerprinting methods for crude oils, residual oils and polymer additives to petroleum products, preparative scale methods for preparing standards and high priced additives.

A number of papers were published on GC column technology. The idea of describing column performance in terms of resolution and time was used by Ettre (67L) to extend the applicability of the separation value concept previously described. The GC analysis of *n*-alkanes (C₇ to C₁₆) was used to compare performance data for packed and wall-coated open-tubular columns. Hawkes et al. (93L) listed preferred single stationary liquids for GC. Most GC analyses can be performed on one of the following: dimethylsilicone, 50% Ph methylsilicone, polyethylene glycol (mol wt above 4000), diethylene glycol succinate, 3-cyanopropylsilicone, and trifluoropropyl-methylsilicone. A list of 18 other liquids with properties intermediate to those on the primary list was also given. The preparation of stable glass capillary columns with polar stationary phases was described by Blakesley and Torline (24L). The procedure involved cleaning with solvents, acid, and base, treatment with dichlorodimethylsiloxane and silanized SiO₂, followed by a 3% solution of the stationary phase in 10 mL of a suitable solvent. A GE XE-60 column had 437 000 theoretical plates, or conservatively 165 000 effective theoretical plates. Gustavo Ober and Didyk (90L) developed a dynamic coating method for preparing capillary GC columns for the analysis of petroleum hydrocarbons, and compared the performance of these columns with that of packed columns. Martire (155L) derived a general theoretical equation for the infinite dilution molal solute activity coefficient (n_2^∞) in non-electrolyte solutions, from which an expression was developed relating n_2^∞ for *n*-alkane mixtures at 80.0 °C to the solute carbon no. and stationary phase mol wt. The equation was used to study the dependence of n_2^∞ and the retention index for C₆–C₉ and C₂₄–C₃₆ alkanes, benzene, and *n*-pentyl chloride as solutes on the stationary phase mol wt. Pacakova and Ullmannova (174L) studied the effect of the structure of the GC stationary phase on the retention characteristics of various HC's, ethers, alcohols, and carbonyl compounds using surface-coated capillary columns (octadecane, 1-chlorooctadecane, and octadecanol liquid phases). Retention volume, retention index, and differential heat of solution were calculated and compared with corresponding measured values. A patent was issued to Luke and McTaggart (145L) for the separation of naphthene and paraffin HC's in the presence of aromatics via GC. Aromatics were retarded on a 10 X molecular sieve precolumn and the main components of the mixture were separated on 13 X molecular sieves. The aromatics were recovered by back-flushing. Equipment to make the separation was described.

Szeri (218L) described a method of preparing gaseous and vapor standard mixture for GC. An evacuated cylinder is weighed and the components of the mixture are added successively, the cylinder being weighed after each addition. Successive dilution is used when one component is present in very low concentration. Large quantities of multicomponent gas mixtures are standardized chromatographically by comparison with binary mixtures of each constituent in a diluent gas at concentrations approximately that of the sample. Accuracy is reported for mixtures containing up to 7.8% C₂H₆, for 0.1% CO in N₂ and for 0.5 to 4861 ppm benzene or hexane vapor in He.

Frankiewicz and Williams (76L) were able to maintain GC column efficiency with large volume, low pressure gas sampling down to 0.25 mm Hg for compounds with short retention times by using Umstead's injection valve (*J. Chromatogr. Sci.*, 12, 106 (1974)), modifying the secondary He supply by adding a ballast tank close to a 100- or even a 500-mL sample loop, and substituting a single-stage for a two-stage regulator. Little efficiency loss was observed at 0.23–760 mm Hg for mixtures of He and 85 ± 3 ppm (vol.) each of CH₄, C₂H₆, C₂H₄, C₃H₈, and C₃H₆ during injection through 100- and 500-mL loops onto a GC column.

Krimond and Rozengart (129L) described microcatalytic attachments for a capillary GC which allow the determination of the material balance during studies of catalytic reactions in the pulse regime. One version consists of a proportioner into which the carrier gas is fed, a microreactor in an automatically controlled temperature bath, and a metallic tube with an attached syringe needle for introducing the sample. For coke-forming reactions, a more complex arrangement allows the recording of peaks of the product and starting material, from which coke yield can be determined.

Two papers on continuous GC's appeared. Watabe et al. (234L) described a counter-current "continuous" GC for multicomponent systems consisting of 3 columns containing the same packing. The first 2 columns served to separate compounds with small partition coefficients and the last to remove compounds with high partition coefficients. The equipment and theory are discussed in detail and illustrated with various separations, including cyclohexane, benzene, and toluene. Reid (188L) recycled components through a basic GC column length up to three times to achieve greater resolution. Results are given for mixtures of (1) acetaldehyde, propylene oxide, and propionaldehyde, and (2) *m*- and *p*-xylene.

Several papers appeared on the use of computers in GC analysis. Kahn and Gill (115L) discussed GC automation in terms of sample preparation, instrument operation, and data acquisition and handling. The discussion was illustrated with results from analyses of wine and natural gas. Shell Research Ltd. (183L) installed a Datachron 2 computer system to acquire and process data from 12 GC's used for the analysis of pollutants, lubricants, liquid and gaseous and customer service problems. Shefter and Borodavko (202L) determined the composition of gas mixtures containing H₂, O₂, N₂, and C₁–C₇ HC's by obtaining the necessary number of chromatograms (up to 4) and processing the peak dimension data in a Ural-2 computer (2.5-min. computing per sample). The calculation sequence was discussed.

Other Laboratory Methods. There was increased activity in the past two years in the field of LC. Knox (126L) surveyed the state of the art and the future of high-speed LC. His survey covered basic principles, theoretical studies of the flow and mass transfer in fixed beds and important applications including the separation of (1) *o*-, *m*-, and *p*-terphenyls on 20 μ Spherisorb alumina AY with 50% water-saturated hexane eluent; (2) aromatic HC's by π-complexation with picric acid on 20 μ Spherisorb alumina with hexane eluent; phenols on pellicular silica gel with 1% β,β'-oxydipropionitrile and hexane eluent. McNair and Chandler (151L) discussed the selection of component parts for LC, including a solvent reservoir, pump, injection system, columns, density detector and recorder. The features of different commercial models were also noted. Szakasits and Robinson (217L) described the use of a porous alumina disk-shaped conveyor to transport LC column effluent into a solvent evaporator and then into a flame-ionization detector. This procedure significantly reduced back-diffusion of the sample manifested by signal spikes. The detector has a linear response and a minimum detectable level of about 2 ng/s. The design, operation, and repeatability of the

detector are described. Relative response of *n*-alkanes and aromatic HC's is tabulated for different detector conditions, and the separation of 6 polynuclear aromatic HC's is shown. The role of column parameters and injection volume on detection limits in LC was analyzed theoretically by Karger (117L), taking into account detector sensitivity and sample dilution. The method was applied to experimental data for benzene and pyrene using water-saturated heptane as eluent, and results compared with those for GC of butane in methane and benzene in pentane. The larger sample volumes possible in LC compensate for the lower LC detector sensitivity, compared to GC. Zakupra and Chernetskaya (242L) described distillation apparatus for removing solvents from fractions obtained by microchromatographic separation of organic substances with an initial bp of 150°. The device consists of 20 receivers (maintained at 60°) and two fractional distillation units. Unger et al. (229L) determined the capacity factors for benzene, biphenyl, *m*-terphenyl and nitrobenzene, using a mobile phase of hexane containing various amounts of water, on a column of silica modified according to Locke (*J. Chromatographic Sci.*, 11, 120 (1973)), and on a column of Merckosorb SI 60 (10 μm). Capacity factors on the modified column were independent of the water content of the mobile phase, in contrast with the Merckosorb. Alessi et al. (4L) analyzed a catalytic gas oil using LC prefractionation, with butyrolactone on Chromosorb P as the stationary phase, followed by capillary GC with PEG 1500 or Apiezon L as the stationary phase. This method was more effective in separating and characterizing a large number of components than prefractional distillation.

Several papers were published on analytical applications of IR spectroscopy. Griffiths (88L) reviewed IR instrumentation, reference data, computerized spectral search routines, and industrial applications. The latter included methane cracking, acetylene manufacturing, toxic gas monitoring in ambient air, identification of oil spills, studies of surface and catalytic reactions, analysis of engine oils, applications of Fourier transform spectroscopy and future developments in IR instrumentation. Fox (75L) described a computer program that used the file-searching technique for matching reference spectra with unknown IR spectra. Both peak location and peak intensity data were used. A library of over 6000 reference spectra has been generated. The program is aimed mainly at identifying unknown materials, particularly those occurring in commercial products. Computer retrieval of IR spectra by a correlation coefficient method was described by Tanabe and Saeki (219L). The similarity of two spectra punched on two paper tapes was judged by computing a correlation between them. A data set of 110 liquid compounds was prepared. It should be possible to apply the method to a data set of 100 000 reference spectra. Correlation coefficients between the same compounds in mixtures of *o*- and *m*-xylene (1:9) are above 0.95 when spectra in the 1200–650 cm⁻¹ range are measured at 10 cm⁻¹ intervals and when the purity of the unknown samples exceeds 95%. Hirschfeld (98L) used a computer method to resolve the IR spectra of unknown mixtures without isolating the constituents. By computer manipulation of repeat spectra of partially fractionated samples, an array matrix inversion may be constructed that gives recognizable spectra of the mathematically separated constituents. Application of the procedure to a mixture of toluene, cyclohexane, and hexane was described.

The use of MS was the subject of several papers. Casalini (37L) surveyed the operation of the MS and the following applications in the petroleum industry: determination of mol wt or elemental composition of linear and cyclic saturated, unsaturated, and aromatic HC's; resolution of a mixture of polycyclic aromatic HC's in engine exhaust (combined with GC); identification of an unlawful component in commercial gasoline, analysis of paraffins, naphthenes, and aromatics in a Kirkuk gas oil, and a scheme for the complete analysis of heavy ends. Stepina (211L) gave a brief review of PETRO-MASS '73, an international seminar on the use of MS in the petroleum and petrochemical industry, organized by the Technical University of Prague. Twenty-one papers were presented and a program for coordinating the development and application of spectrometric methods was adopted. A computerized fast scanning GC-MS was developed by Hedfjaell and Ryhage (94L). The unit uses a preprocessor to bunch a suitable number of samples from the analog to digital

converter to give an optimal digital filtering effect from different scan speeds. Using a capillary column and a scan speed of 0.7 s for the range of mass-to-charge ratios 5:1 to 500:1 and a repetitive frequency of 1.4 s, good quality spectra were obtained even for GC peaks eluting during a few seconds. Results include μg of a commercial solid paraffin dissolved in μL of hexane. Franzen et al. (77L) found fractionated evaporation of mixtures from a direct inlet system which maintains a fixed total ion current useful for separating mixtures consisting of a few components and taking "clean" spectra from contaminated substances. The process and equipment were described and curves for several mixtures presented, including 1:1:1:1 C₂₂-C₂₄-C₂₆-C₂₈ *n*-HC's and tetraphenylethylene-benzophenoneazene. Anbar and Aberth (7L) reviewed the multipoint field source and multiscan field ionization MS, and their use in the analysis of complex mixtures, including crude oils, fuel oils, and air and water pollutants.

Oshima and Kamitake (171L) described a computer program for searching an x-ray powder diffraction data file, using a PDP-8/L. Good results were obtained with complex standard mixtures. Data are given for the composition of a cylinder head deposit and an exhaust valve deposit from a gasoline engine, and a marine engine deposit.

Selective modulation, a new instrumental approach to the fluorimetric analysis of mixtures without separation was described by O'Haver and Parks (168L). The method permits one to obtain the excitation and/or emission spectra of either component in a mixture of two fluorescent species whose spectra overlap too severely for conventional wavelength selection to be effective. A commercial fluorescence spectrometer was modified to carry out the method. Results show that ppm levels of benz[a]anthracene could be measured in the presence of excess chrysene, or of pyrene in the presence of excess anthracene. A carbazole impurity in an anthracene sample was identified.

Young (240L) patented an apparatus and method for detecting free water in hydrocarbon fuels. The sample to be tested is drawn into an evacuated vial containing fresh, finely ground fuchsia dye, and CaCO₃ having an average particle size of <10 μm and a surface area of 5–8 m²/g. Free water reacts with the vial packing to produce a characteristic color. Lidderman and Sidorov (139L) described a moisture meter for high-boiling (>200°) substances such as petroleum fractions. The sample is introduced into a closed chamber partially filled with packing maintained at 140 ± 5° and the water vapor pressure measured in a simple manometer. Provision is made for air purging after each determination. Range for the method is 0–5 vol % water, determination time is 0.5–1 min and the error is ±4%.

Process Analyzers. Moisture. The measurement of water in hydrocarbon oils and gases continues to be an important development and application area. Bailey (13L) has reviewed instrumentation for on-line moisture measurement including IR analyzers, precision hygrometers, and other types of moisture sensing instruments. The survey covers Panametrics Inc.'s Model 2000; Teledyne Analytical Instruments' photometric unit, Anacon Inc.'s Model 206 IR unit, and Mine Safety Appliance Co., Instrument Division's IR analyzers. Wiederhold (236L) covered the principle, design, operation, accuracy, and application of saturated salt dew point sensors, condensation-type and electrolytic hygrometers. Compounds which cannot be measured for moisture content by the electrolytic method are reviewed.

Schnitzler (199L) discussed the use of the Beckman Trace Moisture Analyzer—Model 340 for determining water contents in natural gas. It gave satisfactory service for moisture contents of 20–30 mg/m³ of a dew point of -45 °C at 1 atm. or -12 °C at 67.5 atm. Traces of methanol and higher hydrocarbons necessitate regeneration every 6 to 8 weeks. Accuracy is not affected if the methanol content is less than the water content and condensables do not exceed 70 mg/m³. Lauffer (132L) discussed the use of the Panametric's Model 2000 Hygrometer and 15M22 sensors on natural gas and enriched cracked gases having dew points of -40 to 15 °C. The system showed an accuracy of better than ±1 °C but after 6 months of storage or exposure of the sensors to methanol or natural gas, new calibration curves were required.

Ovchinnikova et al. (173L) have determined the moisture content of CH₄ automatically using the reaction of moisture with NaH to produce H₂, which has a TC about 6 times that

of the components of natural gas. The TC of the natural gas passed over NaH is compared to that of the predried natural gas. On mixtures containing 1 to 100% H₂O, the greatest deviation from the calibration was less than 1%.

Lineberg (141L) developed an apparatus for the continuous measurement of water in oil. The oil is pumped into a heated chamber to evaporate the water which is condensed in an external condenser coil and then reevaporated in a coil inside the chamber. The water content is determined from the vapor pressure measured at the outlet of the latter coil before an adjustable precision throttle valve.

H₂S-SO₂. Two papers discussed the measurement of the H₂S/SO₂ ratio in the tail gas of the modified Claus process. Robinson (189L) discussed the use of Barton's titrator ratio measurement and control system to automatically adjust the flow of air to maintain optimum operating conditions. Another paper (169L) describes the use of Honeywell's Process GC to measure the ratio and an associated control system to control the reaction furnace air bypass valve.

Oxygen. Oxygen analyzers are preferred by Parnell (176L) over CO₂ analyzers for monitoring the combustion efficiency in boilers. The operation of the Taylor Servomax Ltd. O₂ analyzer which measures the paramagnetism of O₂ in a sample aspirated from the flue by a steam ejector probe is described. Bond (29L) discussed the Kent Instruments Ltd. fuel cell type O₂ probe placed directly in the flue. The probe consists of a Zirconia tube with Pt electrodes inside and outside. Using air as a reference on the inside, the voltage produced between the electrodes gives the O₂ concentration on the flue gas side since the voltage obtained is proportional to the differential O₂ pressures. Accuracy is comparable to the paramagnetic cell. Units used in power plants have successfully controlled oil-fired boilers at 0.5% excess air. Jobe (113L) developed a monitoring device for detecting surfactants in liquid hydrocarbons, particularly gasoline and jet turbine fuels. The timing of the separation of aqueous NaOH drops at controlled flows from a Teflon coated probe in a flowing HC stream ~10 cm³/min) in a 15 cm³ cell is detected electronically. Time for detaching is calibrated against known surfactant contents.

PHYSICAL PROPERTY METHODS

Laboratory Analysis. A number of papers dealt with various aspects of distillation. Two papers concerned the use of GC to obtain boiling point data. Kimball and McCracken (122L) successfully applied GC distillation to quality control in gasoline blending operations. The data obtained can also be used to evaluate the column efficiency of pipe stills and other fractionating units. Correlations of Reid vapor pressure and final boiling point specifications with GC distillation give better reproducibility than those based on ASTM D-68 distillation. The derivation of regression equations for correlating D-86 curves for jet fuel, diesel fuel, kerosene, distillation fuel, and various heavy solvent naphthas with GC distillation data is described in detail. van Zyl and Judd (244L) used a new boiling point characterization procedure to smooth the material balance around a debutanizer-gasoline splitter in refinery performance tests. The required component analyses were generated using a high-speed, low resolution GC to group similar real components into a smaller number of pseudo components, which were quantitatively analyzed and qualitatively identified by their average boiling point. The approach offers a practical compromise between a true boiling point analysis and a component analysis by high-resolution GC. Klesment (125L) combined rectification and GC for the separation of multicomponent mixtures from shale extract or tar according to boiling point. The sample (10–25 mL) was steam distilled (400–700 Torr) through a Vigreux column into a GC column, using steam as the carrier gas. Flask temperature was increased synchronously with GC column temperature but kept 10° to 30° below the latter. The GC column (12 mm × 2.8 m) was packed with Rysorb C (0.3 to 0.4 mm) coated with Apiezon L and temperature programmed at 20° to 30°/h. The separated compounds were condensed at 50 Torr and fractions collected at equal time intervals of 5–15 min. Composition of the fractions was determined on an analytical GC column (2.5 cm × 4 m) packed with 5% Apiezon L on Chromosorb G, heated to at least 100°. Separation of compounds with boiling points up to 400° was possible.

Zanker (243L) published a nomograph which reduces the lengthy calculations for determining the fractionation effi-

ciency of packed lab distillation columns by ASTM 2892-73, involving distillation of a test mixture of 30% heptane and 70% methylcyclohexane, and measurement of η_{residue} and $\eta_{\text{distillates}}$. A magnet, mounted on the apex of the sensing tube, is driven by an electromagnet at the resonant frequency of the tube. When the tube is filled with a liquid, the vibration frequency international standard specifications. Vapor temperature is measured with a resistance thermometer. Distillation curves are recorded or printed. Ivan et al. (112L) described an improved distillation apparatus for Kjeldahl N determination. Operation is very simple, the steam supply can be easily controlled and contamination is minimized by using glass or plastic surfaces. The equipment is of simple modular construction to facilitate repair or replacement.

Aggarwal et al. (2L) described an automatic 4-stage vertical-type zone refiner for the purification of organic materials (e.g., naphthalene) with melting points between 50° and 300°.

A new type of density meter was described by Persinger et al. (178L). The sensing element is a V-shaped glass tube with the apex up, passing through a flange to which it is anchored. A magnet, mounted on the apex of the sensing tube, is driven by an electromagnet at the resonant frequency of the tube. When the tube is filled with a liquid, the vibration frequency decreases and the driving electromagnetic circuit changes to match the new frequency. The time required for a set number of vibrations is measured with a solid-state clock and converted into relative density. Sample size is less than 1 mL. The time count is displayed in less than 1.5 min and is available in code form for transmission to a computer. The precision is $\pm 1.5 \mu\text{g/g}$. The meter can be used as a flow-through detector in LC in some instances.

Gleissle and Reichert (83L) described a rotational rheometer which measures the shear stress function and two normal stress functions in highly viscous liquids at shear rates up to 10 000/s and temperatures up to 260° within 10 to 100 ms. The reduction in shearing time resolves to some degree the difficulties in determining these rheological properties caused by the considerable heat production in the flow. Results obtained with a highly viscous polydimethylsiloxane showed much less structure breakdown during acceleration than in a cuvette apparatus. Drislane et al. (63L) discussed the design, operation, accuracy, sensitivity, and response time of the Instron Corp. rotary rheometer. This unit is capable of making measurements of shearing viscosity and first normal stress difference under steady, sinusoidal, or other time-varying input conditions. It can also be used for eccentric disk measurements of the real and imaginary parts of the complex modules of elasticity for a wide range of materials from polymer melts to dilute solutions. Results are given for two silicone fluids and a solution of polyisobutylene in cetane.

Cook and Tock (51L) described the use of aqueous membranes for the separation of pairs of the gases N₂, CO₂, O₂, propane, and ethane. The membrane films were prepared from 2% aqueous solutions of the surfactants Duponal WN or Ivory Liquid. Permeation constants were determined for each pair of gases and compared with theoretical values. Enrichment of one component in mixtures of CO₂ with propane or N₂ could be achieved by establishing a thermal gradient across a membrane of Ivory Liquid.

Hummel (105L) received a patent for an apparatus for the analysis of a gas mixture based on measuring the electric or magnetic susceptibility without using a reference gas. A cuvette containing the gas (flowing or at rest) is placed in an alternating field, and an alternating gas pressure is developed and measured directly by using a receiver and converted to an electric signal giving a measure of the susceptibility. The apparatus was used to measure SO₂ in SO₂-N₂ or SO₂-air, and NH₃ in NH₃-air mixtures. The apparatus can also be used as a GC detector in hydrocarbon analysis.

Evans (68L) reviewed commercial instruments available for surface and thin film analysis.

Process Systems. Several physical property analyzers were discussed the past few years. The major properties covered were viscosity, density, flow, pressure, level, octane number and some miscellaneous properties.

Viscosity. Oppliger et al. (170L) developed a new technique to measure on-line low viscosity liquids. A modified oscillating cylinder with a torsional pendulum vibrating at its natural frequency provided a stable and accurate transducer for

measuring changes of less than 0.1% in the 0.1 to 100 cp range. The transducer with its crossarm, sheath, stem, and sensing tip has two resonant frequencies in the plane perpendicular to the main axis. Neither solid particles nor high flows affect its sensing ability. Its use on kerosene is discussed. Putzker (186L) discusses the rotational coaxial cylinder system for monitoring or controlling viscosity-dependent processes. The design and operation of the Process Viscometer Type KS and KD for measuring viscosity of liquids in storage tanks and pipelines and their application in polymerization processes, oil blending, and other type processes are given. Hoek (100L) explains the development and use of a fuel oil viscometer, Visc 21, which is used in land and ship installations. The unit controls the preheating of oil to a viscosity set point for a particular furnace to provide optimum combustion characteristics. Less burner fouling occurs with better combustion at low-excess-air operation, even when changing from one oil to another. Scheve et al. (195L) described a simplified continuous viscometer for non-Newtonian fluids. The prototype consists of 2 pipes of slightly different diameters connected in series. Axial pressure gradients and flow rates are measured. These values are mathematically related to shear stress and rates of strain at the fluid-pipe wall interfaces. Data are given for glycerol and starch paste solutions as test fluids. Results agree with shear curves obtained by classical capillary analysis.

Density. Picker et al. (180L) have developed a digital readout flow densitometer for liquids. The unit has a V-shaped stainless steel tube of 0.030-inch i.d. soldered in a brass plate. The resonant frequency is sustained by a magnetic pick-up attached to a rigid support. The natural oscillation of the tube is measured at the output of the operational amplifier by a digital frequency meter with a 100-ns resolution. Since the period is 2 ms, a precision of 1 part in 10⁷ is required and obtained by averaging over 10⁴ periods or ~20 s. The filtered liquid is fed to the tube at a flow rate of 0.5 cm³ per min. Each experiment takes 5 to 10 min. The instrument can be used in quality and process control kinetic studies and in series flow calorimeters. Kulakov et al. (131L) describe a flow densitometer using a flow-through vibrator tuning fork device. The frequency of the fork changes with the density of the liquid flowing through the unit. The frequency is converted to a dc voltage and recorded. Temperature changes are compensated automatically. Minges et al. (159L) have developed a hydropneumatic apparatus for continuous density measurement. The level of a partially immersed hydrometer float is kept constant by regulating the liquid level in a float chamber through which the liquid whose density is to be determined flows continuously. A bubble tube follows the hydrostatic pressure in the vessel and a linear relation is calculated between the density and the pressure. The sensitivity is determined by the dimensions of the float. To amplify the pressure changes in the bubble tube, elements of low-pressure pneumatics are used to operate valves determining the liquid level.

Exxon R & E Co. developed a new system (43L) for determining the density of LPG and LNG in large storage vessels. Capacitance probes immersed in the liquid determine the dielectric constant and determine the temperature and density. In a 400 000 bbl LPG storage tank, density was measured to within 0.1% of its absolute value, which is better than other techniques.

Flow. Bailey (14L) reviews various type flowmeters including those based on fluidics, solid-state diffused silicon pressure transmitters, variable reluctance, positive displacement, magnetic, differential pressure, vortex shedding, turbine meters, differential capacitance, and ultrasonics. Lock (144L) discussed the Fisher Porter Ltd.'s new Mag-X electromagnetic flowmeter using intermittent dc current which gives high accuracy in low pressure use, simpler installation, and a stable zero. Applications include facilities for water and waste treatment, paper processing, acids, alkalies, and hydraulic turbines. Dawson (57L) compares various flow measurement devices and their operating principles and calibration, including turbine, vortex, and orifice meters of the differential pressure type, venturi tube, and target types. Mentioned also is their suitability for use in advanced electronic systems. Guenther and Rodely (89L) describe a mass flow measurement system for LNG. It consists of a vortex-shedding volumetric flowmeter, a capacity density transducer and a

computer. The flowmeter pulses enter a digital scaling circuit assuming the density of the LNG to be at its maximum value of 35 lb/ft³. Another digital scaling circuit controlled by the density signal of 0–5 V blocks a portion of the pulses in a manner proportional to the density. The resulting pulse output is proportional to the total weight of LNG at the operating density. Pavlov and Polyakov (177L) review contactless flowmeters for liquids based on continuous NMR observations and gives 19 references.

Pressure. Lawford (134L) surveyed the application of differential pressure instruments in over a dozen specific applications such as viscosity, density, leak detection, level, flow, and in various protection devices for centrifugal compressors and uses in filters, distillation towers, and other processing equipment. Votava (233L) surveyed mechanical transducers which convert measured pressure to displacement including diaphragms, bellows, Bourdon tubes, and straight tube and resistive transducers. Also included were capacitance, pressure, and inductive transducer elements such as piezoelectric crystals, vibrating cylinder and diaphragm elements, null balance and force balance units.

Level. Geake and Smalley (81L) describe optical depth gauges and level controllers for liquid such as fuels based on work done at the University of Manchester Institute of Science and Technology. Specific applications and operating principles and limitations are given. Shunta (204L) discusses the use of a proportional sampled-data controller for designing a dead time level-control system as might be used in a distillation column. Charts show controller gain settings for a given damping coefficient as a function of process parameters. Charts are also given to size vessels based on maximum volume change occurring after a maximum step change in volumetric flow. Anderson (8L) gives a simple equation which relates liquid surface area, dynamic system gain, valve capacity, controller proportional band, and level sensor spans. Based on the equation, the sizing of control components can be made. A useful rule is that the ratio of maximum flow rate to the liquid area should not exceed 4.5 in./s. A different article (167L) describes a special sonar sensing head that pinpoints fuel oil levels. The "sonargage" uses a fluoropolymer for the sensing head and the sonar principle to measure distance between a fixed point and a varying level. The sensing unit and the electronics are connected by a coaxial cable carrying less than 2 watts at 12 V and is packaged for Class I, Division I, Groups A, B, C, and D areas as long as the electronics are in a safe area. Readout can be remote.

Najera et al. (164L) used a radioactive device to control the interface level of a pilot DEMEX unit to prevent solvent loss and maintain resident time. The device consists of a cesium-137 source in a stainless steel capsule mounted at the required level on one side of the extractor, the detector on the other side, a recorder to indicate the difference in densities between the two phases, and a control device. The advantages of the system over other level detection systems and shielding against radiation leakage are discussed.

Octane Number (O.N.). Fenske and Pasik (70L) describe an apparatus for determining fuel composition and O.N. of HC's. It consists of a thin-walled combustion chamber within a heat exchanger bath, with inlets for O₂ and fuel to produce a stabilized cool flame. A moveable temperature probe consists of a pair of thermocouples which can be positioned to indicate the location of the cool flame and, with a controller, can regulate the combustion parameters to position the flame at a predetermined point. The fuel inlet is tied to a sampling inlet system for the material being analyzed. Clinton and Puzniak (47L) describe Gulf's on-line Process O.N. Analyzer. The unit consists of a temperature-controlled and explosion proof reaction chamber with microthermocouples to measure differential temperature, a programmer for sequence sampling, a signal conditioner, and a recorder for O.N. The system operates on the basis of the correlation of the induction period and the magnitude of the exothermic temperature peak with O.N. ratings. Two years of testing show it to be as accurate as the standard ASTM method with improved reliability and lower maintenance. It can be used on various gasoline product streams and in blending gasoline components. In another paper, the same authors (46L) discuss the evaluation of on-line O.N. analysis employing the cool flame oxidation technique. The reproducibility is ± 0.5 . O.N. Applications include FCC and alkylation units and other gasoline component producing

units.

Miscellaneous Physical Properties (Temperature, Refractive Index, Specific Ion, Radiation, Weight.) Hemardinquer (95L) reviewed the operation and characteristics of various temperature measuring instruments and temperature controllers. Sandford (194L) discusses temperature monitoring and control with emphasis on new developments in temperature sensors of increased reliability, accuracy, and ease of interfacing with controls. The review includes the operation, accuracy, response, and characteristics of resistance temperature detectors, use of thermocouples in tubes in oil and gas fired furnaces; thermopiles, thermistors, IR temperature sensors, the temperature range of various sensors, and their manufacturers.

Schmid (198L) has developed a refractive index unit using several curved optical fibers of graduated RI and high transparency. A photodiode measures the number of fibers of higher RI than the fluid. Geake (82L) has developed a critical angle refractometer using a spherical concave or convex optical surface to give a linear RI scale. The unit can be inserted via flanges in a pipeline as an obstruction free unit. An extended face model can display the variation of RI with depth in a tank or monitor mixing processes or stratified flows.

Hunt (106L) reviews the use of specific ion measurement for process control. Response times under ideal conditions are rapid but the presence of interfering ions, low activity levels, and poor mixing or flow may slow response from a few seconds to 5–10 min. The advantages and disadvantages of solid state and membrane electrodes are included.

Bond (26L) gives a description of analytical measurement systems of the noncontact type involving β or γ radiation from radioisotope sources. Uses include level measurement, belt weighing, density, moisture in solids, sulfur in HC's. Manufacturers of various units are given.

Dennis (61L) describes the operation, advantages, application, and costs of electronic weighing systems. Uses are varied including inventory control, monitoring of materials used in oil fired electric generating stations or large scale industrial furnaces.

IMPROVED INSTRUMENTATION AND TECHNIQUES

Laboratory Analysis, GC Methods. The bulk of the work reviewed in this section involved some aspect of GC.

Thiede and Ehrlich (221L) described a commercial dual column digital GC. FPD, FID, and electron capture detectors can be used. The electrometer has a dynamic range of 6 decades. The calibration curves with a square-root function are linear over 3 decades. In the determination of 17 components in a petroleum distillate, the relative standard deviations were $\leq 2.6\%$.

A number of papers were published on various aspects of GC column technology. Delventhal et al. (59L) investigated chemisorption GC with phosphinodithioate complexes. Polymeric and monomeric phosphinodithioates with Ni, Co, Pt, or Pd were used as stationary phases, supported on Chromosorb W AM-DMCS for the separation of alkenes, ketones, amines, organic phosphates, thiols, sulfides, and thiophenes. A procedure for making high efficiency Cu capillary GC columns was described by Le Chi and Sakodyniskii (135L). The column (0.25–0.30 mm \times 50 or 100 m) is blown out with dry N₂ at 4–5 atm. for 1–2 h, washed with methylene chloride and ether, dried several hours with N₂, treated with K₂Cr₂O₇–H₂SO₄ solution and washed twice with each of the following: EtOH, ether, acetone, and hexane, with drying after each washing. The column is coated with Apiezon L or squalone in solution, with the addition of Span 20. The total amount of a phase should not exceed 2–4 mL, and filling should take at least 3 h. The coated column is held at the maximum operating temperature for as much as 8 h for stabilization. Polar phases require O₂ treatment. Ayers and McCoy (10L) patented a GC separating system with 6 columns and 2 ovens or temperature zones. The columns are arranged in 4 sub-systems with 3 columns in one temperature zone and the remaining 3 columns in the other temperature zone. Portions of the sample are fed to each sub-system or stream and selected components are detected in each of the 4 streams. A patent was issued to Hermann and Sasse (96L) for an apparatus and method for temperature-programmed GC using

adsorbents. *N*-Alkanes (0.3–23.6%) were determined in 2- μ L samples of HC distillates and deparaffinized oils using a programmed temperature (275°–600°/min) on molecular sieves. The determination of 23.6% *n*-alkanes in a middle distillate required 3 min. Reese and Grushka (187L) discussed a novel method of packing preparative columns for GC. The packing is pulled upward into the column with vacuum, the bed homogenized, and the column turned over and tapped on the floor. The process is repeated until the column is filled. The efficiencies of columns (3.3 cm × 86 cm) packed by this procedure equalled those of analytical columns with HETP 0.5–0.7 mm.

Several papers were published on the application of computers to GC column technology. Joensson and Joensson (114L) measured GC retention volume directly with an on-line minicomputer. The V-shaped column was heated in an oil bath to 60° with temperature control accurate to within 0.01°. Mass flow of the carrier gas was measured with a thermal mass-flow meter. Outlet pressure and pressure drop in the column were measured with capacitative pressure sensors and signals from the FID were amplified. Each of these parameters was measured simultaneously by the computer at intervals from 0.05 to several seconds, depending on peak width. An integrated volume was calculated at each point on the chromatogram. The retention volume of each peak was calculated as the first moment (Grushka et al., *Anal. Abstr.*, 19, 1892 (1970)). The sample used was heptane and the column stationary phase was 20% octadecane. Lekova and Gerasimov described a computer method for the selection of a suitable stationary phase in GC in two parts. Part I (136L) discussed resolution criteria based on relative retention times. They propose that relative retention data for selected components of a given mixture be determined on a number of stationary phases. The degree of separation *R* of two adjacent compounds is then determined with the use of a computer. The most suitable phases for the separation are those giving the largest values of *R*. The method was applied to the analysis of a mixture of HC's on HC and silicone stationary phases. In Part II (137L), a mixture of HC's is separated by GC using 37 stationary phases. The separation of the components obtained with each phase is calculated according to a formula given in Part I. The data are fed into a computer in ALGOL algorithmic form, from which are selected the three most suitable phases for the separation.

Several publications on GC sampling appeared. Mlejnek and Blatnický (160L) described an adapter for commercial external injection GC inlets. The unit has a stainless steel tube in which a sample-containing ampule is placed, a plug with one end inclined and an eccentric bore and a stainless steel plunger with an end inclined to match that of the plug. Holes are provided in the tube around the plunger for carrier gas entry. A silicone rubber ring seals the plunger end. A patent was issued to Valentin and Hagenbach (231L) for an apparatus for injecting a liquid sample into a GC column. The apparatus provides precise injection with distinct starting and cutoff times. The sample is stored in a tank under pressure. An automatic precision valve injects the predetermined amount into a flash evaporator that is constantly swept by carrier gas, and is directly connected to the column. When the sample is injected, the valve closes and carrier gas sweeps the short piece of tube between the valve and evaporator. Houghton (103L) described a sample trapping and re-injection technique for GC. The trap consists of a thin-walled stainless steel tube, whose center section is packed either with glass wool or a suitable coated support, attached to the GC column outlet. For re-injection, the trap is connected to a syringe needle. The sample-containing portion of the trap is cooled with a jacket containing liquid N₂, carrier gas is passed through the assembly and the needle inserted through the GC septum. The trap is then heated with a jacket at 220°. This technique gave sharp, nearly symmetrical peaks with HC samples (C₁₁ to C₁₅) without loss of resolution compared to the first separation. Carbonyl compounds (μ g quantities) were also trapped and injected successfully with the technique. Umstead (227L) described a sampling technique for sub-ambient pressures in GC. A valve with a large sample loop (up to 100 mL) was used for dynamic gas sampling at pressures \approx 2 Torr. During injection, the sample is compressed at one end of the loop by a secondary He flow. For a Poropak T column (80–100 mesh, 0.375 in. × 6 ft), the number of theoretical plates is re-

duced from 830 to 234 for methane, from 920 to 880 for propane. Kindsvater and Rietz (123L) described a simple collector for trapping air-sensitive high-boiling effluents from GC columns. The glass trap described comprises a manifold with up to 8 branches, each consisting of a heated U-tube with a capillary tube gas vent and a 2-mm stopcock. After being flushed with He, samples are collected in the desired tubes, which are then evacuated and sealed off from the manifold. The traps can be adapted for MS or NMR analysis.

A number of papers appeared on GC detectors. Morris (162L) reviewed the following types of detectors for GC and LC: FID, TC, FPD, chromatographic, electrolytic conductivity, UV, fluorimetric, refractometric, heat of absorption, and solute transport. A simple current stabilizer for TC detectors was described by Svestka and Nicolajenko (214L). The unit was checked on glass katharometers equipped with 4 Pt wires (20 Ω each). Stabilized current was 250 mA (\pm 0.1%). Two patents were issued to Bednarski (19L, 20L) for a novel type of GC detector. The effluent from a GC column was split into 2 unequal streams, the minor portion being used for a conventional chromatogram. The major stream was sprayed through a nozzle onto an Al carrier foil strip, coated with Al₂O₃ or silica gel, and moving synchronously with the chromatogram sheet. The components in the mixture can thus be directly matched with the chromatogram peaks and may be identified by techniques such as UV, IR, or extraction.

Afanas'ev et al. (1L) described a pyrolytic microdoser for an automatic control GC system for determining the composition of polymers and heavy petroleum products.

A continuous-surface GC with a 12-in. rotary chromatographic disk channel was described by Sussman et al. (212L). Experimental separations of methane, propylene, and ethylene were compared with computer models, which can be used to determine the optimum conditions for such operations. An equation was derived relating operating conditions to detector sensitivity.

Bartoli et al. (17L) discussed the automation of GC data collection and analysis using a medium sized on-line computer. A computer program and a simple interface were developed. The program allows for variable background subtraction and resolution of overlapping peaks. The hardware and software were described in detail. Programs for spectrum classification and screening of GC-MS data on a lab computer were described by Gray and Groenneberg (86L). Structural features of the compounds expected to be present were assigned based on empirical rules provided by the program. Little knowledge of programming is required, but the analyst must know the types of compounds that occur in the samples and be able to recognize their spectra. Identification information for the sample constituents is printed during the analysis. The programs were used in screening for sterane-triterpane mixtures in oil shales for fatty acids and sterols in fresh water sediments, for the phenolic acid and hydroxy-acid constituents of cutins, and for pesticide residues.

Laurent (133L) discussed simulated distillation of organic compounds of low volatility by means of forced evaporation, where fractional distillation is achieved in a tube through which an inert gas is passed and whose temperature is programmed. The temperature of vaporization is much lower than the boiling point at \approx 1 bar. The equipment is described and illustrated by the separation of a mixture of saturated, straight-chain HC's (C₁₈, C₂₂, C₂₆, C₃₁, C₃₆, C₄₀, and C₄₄). The sample (0.3 μ L) is injected into a stainless steel tube (1-mm i.d. × 200 mm) using a N₂ carrier gas (40 mL/min initially at 1.8 bar) and temperature is increased from 65° to 508° at 20°/min. Constituents in the effluent gas are detected with an FID. The sensitivity is sufficient to distinguish between the oligomers in a sample of a poly(alk-1-ene).

Other Methods. Bombaugh (25L) describes a novel method of generating multislope gradients for high-pressure LC. An exponential-distillation flask is converted to a versatile programmer, capable of delivering linear, concave, or convex multislope gradients and isocratic carrier blends by adding a balanced, by-pass flow system. With this by-pass line, a carrier is added to decrease the concentration of B carrier in the mixing chamber. The flow is controlled by a 10-turn vernier metering valve. Blends containing 10–100% B can be programmed. Essigman and Catsimpoolas (66L) described a simple derivative mode detector for LC which was obtained by a simple modification of commercially available UV

dual-channel detectors. The modification involves a change in the flow pattern of the column effluent in the 2 optical channels. The resulting derivative signal provides information for convenient and precise determination of peak parameters (retention time, standard deviation of the concentration distribution) which are needed for the estimation of the plate height, resolution, and other chromatographic factors.

Martin (154L) described a versatile system of miniature TLC. It is used for a wide range of separations including initial method development and fast quantitation of some components in small groups of samples by non-instrumental techniques. A scaled-up system is also described, which is used for the final development and testing of all methods as well as the fast quantitation of multiple components in large groups of samples by instrumental and computerized techniques. Applications for plant stream control, product specification tests, and rapid quantitation (petroleum-derived detergent-range olefin sulfonates) are discussed.

An off-line digitizer system for spectrogram analysis was discussed by Bukreev et al. (34L). The system consists of a hand-driven scanner (converted X-Y plotter), a multiplexer, and a circuit for driving the digital recorder (punched paper tape). The digitizer system is fed a standard spectrophotometer recorder chart, and the analog record is converted into the punched tape record. The punched tape spectrograms are then analyzed with a computer to yield the characteristic band intensity, harmonics, etc.

Unger et al. (228L) advocate the use of dismountable fluoroplastic ampules (instead of glass or quartz) to achieve more rapid filling with viscous petroleum products prior to EPR analysis. An open ampule is easily filled with a vacuum pump without heating and dissolving the samples. Coates (49L) discussed the use of laser Raman spectroscopy in the oil industry as applied to polymeric additives, synthetic and HC-base oils and low molecular weight HC's. Sampling techniques, purification methods, sample and solvent effects, and calibrations were studied.

A turbulent flame liquid-fuel burner for flame photometric analysis was described by Korovin et al. (127L). The burner uses compressed air to atomize the fuel. A stable smokeless flame is obtained with a wide range of liquid fuels including jet fuels. The fuel is used as solvent for both standard and sample, eliminating the effects of variable composition and increasing the accuracy. The sensitivity and reproducibility for Cu, Ca, Zn, Mn, and Pb using either the liquid fuel/air burner or the normal gas/air burner are similar.

Several publications on distillation appeared. Linder (140L) described a device for sampling petroleum "bottoms" at high temperatures and reduced pressure. With slight modifications, the equipment can be used with high pressures. Glukhov et al. (84L) discussed a laboratory fractionating column designed for automatic batch operation. The column has 5 packed sections, is surrounded by 3 glass jackets and section heaters, has a boiling flask heated with IR, and is controlled by a reflux optimizer with an adiabatic controller, a section-heating controller, and a pressure controller. Components with a boiling point difference of 4° can be separated and distillation analysis can be done with an accuracy of 0.1°. An apparatus and method for automatic determination of the boiling point was patented by Fourre et al. (74L). The equipment is suitable for determining the boiling point curves for crude oil, petroleum fractions, and other HC mixtures. The sample (150 g) is first distilled at atmospheric pressure to 200°, followed by vacuum distillation at a preselected pressure until a preselected temperature is reached. The vacuum temperatures are automatically converted to temperatures at atmospheric pressure and the atmospheric boiling points are automatically plotted vs. % overhead. Stary (210L) described a laboratory apparatus which may be used for controlling temperature, monitoring distillation column reflux, carrying out temperature programs in GC, monitoring zone refining, etc. Electronic time relays are interconnected into an uninterrupted cycle so that they form a sequence of 3 independent time-variable electronic pulses, which are repeated cyclically. These pulses are transformed into mechanical motion in an evaluation section of the apparatus.

A new instrument for measuring the gas permeability of porous materials was described by Czolbe et al. (52L). The measuring principle is the determination of the time for the passage of a specified inner volume through the sample at a

specified pressure difference. The air volume is determined by displacement of water into a vertical container consisting of a stack of 4 cylinders, increasing in size to the top, with sensors to indicate the start of 1, 10, 100, 1000 mL. Pressurized air is fed through a proportional controller.

Rybnikar and Klepal (191L) modified a penetrometer to provide automatic recording of temperature-deformation (or expansion) curves. The apparatus senses and records the deformation with the penetrometer needle via an inductive amplifier connected to a differential transformer. The temperature is measured by an Fe-Co thermocouple.

Sieben (206L) developed a recording viscometer with oscillating capillary tubes. Two such devices, mechanically coupled, comprise a bridge viscometer with which two fluids can be compared directly. The instrument has a wide dynamic range and is readily adaptable to automatic systems. The capacity of each arm of the viscometer bridge is less than 100 mL. The fluid under test may be allowed to flow through the system.

The determination of salt content in petroleum and petroleum products using the frequency dielectric constant measuring method was discussed by Benin et al. (22L). Salt concentration covered was 0–50 mg/L or 0–500 mg/L after dilution of the sample. A mathematical solution of the problem was given.

Brubaker (33L) described data processing and simulation techniques for chemistry instrumentation systems. The following investigations were discussed: rate distortion theory for the dynamic bandwidth compression of chemistry data; use of digital hardware to reduce error due to finite-length arithmetic in digital signal processing algorithms, digital and hybrid simulation methods to aid in evaluation of data processing and control systems; the teaching of courses on methods of signal processing to chemistry personnel. Robinson (190L) described applications of computers in chemistry including on-line processing of data from laboratory analytical equipment, control of chemical plants and unit operations, simulation in plant design and molecular structure, literature searches, data storage and retrieval, and operational research and management functions. Kalvoda (116L) discussed the functional principles of digital computers, programming, use in chemical instrumentation, and the function and design of the interface in on-line applications.

Dowd and Monkman (62L) discussed some uses for FET input operational amplifiers in analytical instrumentation. Seven circuit designs were presented: a differential voltage and current sensor, an air-earth current sensor, a 10-min time constant low-pass filter, a decade time marker, a complementary voltage tracker, and a 1000-volt sector sensor for MS. Direct signal transmission can be obtained with as much reliability as can be obtained from the vacuum tube.

Process Instrumentation. Major areas covered in this section are process GC, other analyzers, control systems, and computers including microprocessors.

Process GC. A review by Villalobos (232L) of process GC units is given along with the basic elements of the technique including sampling valves, programmer-controller, column design, calibration, and recorders. Interfacing several GC's to a minicomputer which controls each GC and processes all data is considered. Applications discussed include the determination of dissolved gases in transformer oils, vinyl chloride in air, water in xylenes, and HC's in steam condensate. McCreadie (147L) reports on column switching techniques for process GC's. Discussed are 6-port valves, column stripping to remove undesirable contaminants, bypass techniques to reduce analysis time, heartcut methods to analyze trace components on the tail of a major peak, regrouping, and flow switching methods. Bond (27L) discusses a process GC that is adaptable to a specific application. The Servomax 400 series has several options from which to select the appropriate GC design. Options available are: single or multistream and multicomponent analysis; detectors of the cross section, microkatharometer, or explosion proof FID type; detector head amplifier for retransmission of a 0–10 V signal; and special systems for preconversion of a species such as CH₄ to CO₂. Basic components include solid state temperature control, temperature dial indicator, purge air controls, and oven over-temperature cutoff. According to Foxboro (44L), its all pneumatic GC analyzer can be used in hazardous areas and has been successfully used for closed loop control. The unit

automatically samples, analyzes by GC, and transmits concentration signals by 3–15 psig signals. Applications are discussed for controlling distillation towers, monitoring chemical reactions and ammonia plants for H₂ and N₂. Lipavskii and Berezkin (142L) review the problems of the application of process GC in the research and control of petrochemical processes. The dynamic properties of the analysis system are given. Pomerants and Simongauz (182L) discuss the use of a new GC model for automatic analysis of C₁ to C₆ HC's at 2-min intervals along the well bore. The unit features a FID detector, temperature-programming and high sensitivity. Bader (11L) describes a GC method for detecting HC's and N₂ in the O₂ fraction of air separation units.

Other Analyzers. Two papers were given in the LC area. Hoelzgen (101L) describes a fully on-line LC system called Optichrom LC, which automatically takes process stream samples and injects them into a separation column. The equipment can be used in liquid-solid, liquid-liquid, and gel-permeative chromatography. An article (39L) describes DuPont's new Model 480 process LC unit. It should find use in the analysis of phenols, polycyclic aromatics, dyes, pesticides, plasticizers, etc. It handles up to 10 compounds in 4 auto-selected sample streams. Readout can be a complete chromatogram or bar graph or trend. Typical analysis time is 5 min, and recycle time can be set up to 999 s. A photometric detector is used over the wavelength of 214 to 578 nm.

A few papers discuss IR and other spectroscopic analyzers. Dunlap and Henebry (64L) describes the use of the "Pro-sensor", a single beam dual wavelength IR analyzer. Some uses help in saving energy and include the detection of methane slippage in hydrogen furnace effluent; C₂, C₃, C₄, or CO₂ in refinery fuel gas; ethylene in isobutane, butadiene in alkylation feed, and other light HC processes. Aleininkov et al. (3L) derived equations to determine the relative analysis error by a double beam noncompensating IR gas analyzer where one beam passes through the sample cell and the other beam passes through both the sample cell and a standard cell containing a known amount of the gas to be determined. The error caused by temperature and pressure changes in the sample to be analyzed was calculated. When CO was determined in the presence of H₂O, NO, CH₄, and CO₂ over the concentration range of 2 × 10⁻⁸ to 2 × 10⁻⁶ g per cm³, an error less than <0.5% in the log ratio was observed with a relative humidity change from 50 to 98%. Tiley (223L) presents a new approach to obtain a windowless sample system for on-line analysis of liquids, slurries, and powders by x-ray and IR techniques. Fluid jets issuing from silicon nitride orifices overcome many of the problems of sampling with the use of a flow cell with a thin window. Results of tests including paraffin oil doped with S⁰, water in oil, and water in paraffin are given.

According to Perkin-Elmer Corp. (38L), its model MGA-1150 multigas MS process monitor has a response time of 99% in 2.5 s and remains calibrated for months. It can sequentially monitor up to 16 process streams measuring 8 components in each stream. Typical gases detected are O₂, N₂, CO, CO₂, A, C₁, C₂, SO₂, and H₂S. The system automatically introduces a 1-cm³ sample into a dual filament ion source. The cations formed are focused into a beam and split into 8 sections on a mass/charge ratio which ranges from 2:1 to 136:1. Amblard (5L) devised an automatic MS for analyzing effluents from steam or catalytic cracking and refining units. An apparatus allows measured doses of the vapors to enter the MS automatically and continuously and uses circulation systems and valves to permit rapid sampling and analysis. Harman (91L) describes an in-line monitor to detect metallic debris in recirculating oil lubrication systems. The analyzer uses XRF and was designed for aircraft transmissions and gear boxes. Iron in solution and particles up to 300 μm were used for both dynamic and static tests with MIL-L-7808 oil. Both debris mass and rate-of-change outputs are provided. An apparatus is described (30L) for detecting gas leaks in pipes or containers which uses an MS for the final detection of gaseous hydrocarbons. The system has a gas receiver, a converter to dissociate the gas into two parts of which the first part has a molecular weight less than a predetermined value, a cold trap for removing molecules of molecular weight greater than a given value and the MS to analyze the first part. Barre (16L) provides a discussion of nondestructive testing in refineries. Methods are covered for the measurement of pipe thickness by γ rays and ultrasonics, equipment leak detection, surface

temperature and vibration, and other methods to determine the safety of equipment.

Several papers dealt with calibration, maintenance, and testing analyzers. Topham (225L) discusses the code of practice for calibrating process analyzers under consideration by the IP Standardization Committee. The code should assist the manufacturer in substituting precise instrument methods for the ASTM, IP, and other accepted procedures, and will include documents on the calibration of viscosity flash point and other key properties. A similar article (108L) discussed the code of practice for calibrating process analyzers for measuring the quality of gaseous and liquid petroleum products developed by the Institute of Petroleum (IP) Panel ST-L-2 on Process Analyzers. The guide covers initial factory calibration by either reference-sample or paired-sample method, in-service checking and supervisory checking using control charts. It is intended for persons directly involved with supplying, using, and maintaining such analyzers. Hillis (97L) looked at possible ways of improving analyzer maintenance. Factors considered were: proper engineering, particularly sample system design, maintenance organization, training, maintenance records to show how well the system is performing to identify unreliable components, and to determine the frequency of calibration. Flanagan (72L) described the cooperative instrument evaluation performed internationally by the U.K.-based Sira Institute Evaluation Panel (Sirep) with the collaboration of instrument manufacturers. Sirep found 20.5% of 103 instruments from 1971–75 were outside specifications under reference conditions, 29% were outside under influence conditions, 25% had component failures, 22% were received faulty, and 25% had inadequate manuals. Some 34% required modifications in design based on the evaluations. Types of instruments evaluated are tabulated.

Control Systems. Several papers appeared relating to control systems or to sensors involved with control systems. An article (156L) describes Beckman Instruments Inc.'s full line of advanced electronic controllers, computing modules, recorders, transmitters, and accessories designed specifically for the chemical, petroleum, and petrochemical industries. A combination of analog and digital controllers gives "bumpless" operation. New 1-, 2-, and 6-channel recorders use pressure sensitive paper eliminating inking problems. Murrill (163L) looks to the probable 1985 control systems needed for future plant characteristics including more standardization, larger size, more exotic techniques, and more safety and pollution and product quality control, more feed-forward control with fewer plant personnel and the corresponding hardware changes expected. Shinskey (203L) discusses the advantages and disadvantages of positive feedback control. Negative feedback is normally used to provide stable linear response of output to input. However, the reset action in a pneumatic controller is by positive feedback. Examples of negative and positive feedback systems under various conditions are given. The measurement of flare gas is described (65L) using a FM-700 flow sensor which gives accurate readings over a wide flow range and permits steam control in flare pipelines. A pair of thermistors inside the pipe use a correlation factor to determine flow rate independent of gas temperature, composition, and condensation. When a FD-700 density meter is used simultaneously and both signals are fed to a mass calculator, a signal is obtained which can activate the steam input valve. Kostiw (128L) covers the instrumentation of fuel-oil-fired power plant systems with control loop alternatives. Types discussed were: simple temperature loops with and without pressure controllers; temperature loops with steam pressure reduction; viscosity and temperature with cascade loops with either a flow or temperature feedforward signal. Several problems encountered with displacement pumps and the problems occurring when hiring a contractor to design and build a burner management system are given. Belsterling (21L) discusses work done to improve fluidic amplifiers in both signal to noise ratio and dynamic range. Uses of fluidics include: a self-contained electrofluidic angular rate sensor that uses a vibrating crystal pump; gas concentration sensor; fluid strain gauges; diaphragm amplifiers; flowmeters; and aerospace and military application.

Computers—Microprocessors. Several papers relate to the use of computers for control of processes to control of instrumentation, particularly GC's, as well as to the use of microprocessors to handle small analyzer and control systems.

An article (36L) discusses the trend toward increased use of computers, mainly direct digital process control systems; the growing reliability of stream analyzers; new digital devices to handle analog signals; and the use of integrated circuit chips that displace discrete first generation solid-state process instruments. McDevitt (148L) evaluated the use of microcomputers for real-time, on-line adaptive control systems; the hardware and software costs and advantages of adaptive systems for optimizing control strategies to reduce energy consumption; inventory variations, accidents, and product quality. The decreased cost of microprocessors from thousands to hundreds of dollars now makes them available to update control systems and analyzers. Discussed were the design of control systems with microprocessors for digital data gathering, minicomputers for local control, and tie-in to a master computer. Mamzic (153L) surveys the recent developments in pneumatic interfacing equipment including transducing requirements of pneumatic vs. electronic and their advantages and disadvantages, analog controls, computer control stations including sensing circuits and computer to pneumatic converters, and future developments in computer control stations. Shah (201L) surveys the use of minicomputers in conjunction with sensor-oriented monitoring and control. Items covered were hardware and software costs, improved reliability on control of small sections of a plant such as a distillation tower, microprogramming in place of hard-wired commands and peripheral equipment. Bailey (12L) discusses a digital computing controller, particularly the "Micon" which can serve as an all-digital computer and as an adjunct to conventional minicomputer control in a process plant. In one example, an in-line pH control uses a fixed program microcomputer controller to operate a digitized caustic control valve; a feedforward algorithm; and a 3-way valve to divert off-specification effluent. On pipelines, pumping rates can be measured and controlled from digitized degree of polymerization measurements and other digitized signals and permit control of flow and surging in compressors. Bond (28L) describes a hardware and software package for real-time data acquisition, real-time and off-line analysis, and real-time digital control in industrial and research applications. Davis (56L) discusses the different ways instrument companies employ chips. Fisher Controls Co. predicts that microprocessors will impact most in the displacement of analog instruments in complex but well defined operations such as boiler control. Advantages of microprocessor-based data acquisition systems with the elimination of long cables and greater flexibility are given. Several companies offering microprocessors systems are given.

An article (111L) described the application of a computerized turbine engine test setup. The system measured 200 variables and cuts testing time about 5-fold. The system calculates and corrects many of the test parameters to standard temperature, pressure, and humidity.

Stainthorp and Benson (209L) set up mathematical models of a process after selecting the control objectives and variables and operating constraints and solved the formulated process under steady-state conditions. A second program finds the maximum and minimum worse case condition. A third program identifies the steady-state feedforward and open-loop decoupling controllers for better control and a fourth program checks for nonlinear effects. A typical example given was the design of a control system for a finned-tube air heater. Kaufman et al. (118L) discuss the use of a PDP-8 computer system for pilot plant automation that encompasses informing units and GC control plus data flow from these units to a final print-out report for the inquirer. The system has reduced the time from 4 to 5 weeks to less than 2 h and reduced manpower to obtain data and make material balances. The system used 5 GC's, two data loggers for flow rates and temperature, controlled process variables, and scanned alarm conditions. The data reduction used 2 PDP-8's.

Fischer and Macey (71L) describe a digital data-acquisition system to measure engine pressure synchronously with crank angle, including hardware and software. It is a time-shared, user-oriented system which converts analog data to scaled and graphical digital output. Input can be either direct or from an analog tape recorder.

Hobbs et al. (99L) discuss the computer control of a butadiene operation using Applied Automation Inc. hardware. Covered were: input-output systems, language and programs,

control variables in the furfural absorber and stripper, and the butadiene purification tower; control strategies of both hypothetical and actual systems. Improved efficiency figures show a 5% increase in butadiene content and a 36% cut in butadiene recycle.

Schleicher and Dannert (197L) review the functions, advantages, and limitation of 5 interfacing variants between process GC's and computers: (1) computer to one or more GC's via voltage divider and signal adjuster; (2) computer-controlled unit with voltage divider and a variable amplifier; (3) computer-regulated and controlled unit with a voltage divider, variable amplifier, and data reduction and storage; (4) direct coupling via small computer-controlled unit with retention of the GC control unit; (5) direct coupling of GC to a small computer which controls the GC.

An article (185L) describes Bell and Howell Ltd.'s PM 16 microcomputer that can linearize and process a number of physical sensors to give a desired control value automatically. It stores and interpolates difficult custom functions which have no simple definition by equations. A description of the design and operation of the microcomputer, its programming and typical applications are given, including control instrumentation for GC and spectroscopy, inference analysis from indirect measurements such as S in gasoline, and time sharing of small process control loops.

Sutherland (213L) describes a computerized turbine engine test stand and a combustion test rig with instrument systems that measures 200 variables and converts them to engineering units for the operator on a 24-line, high-speed video terminal. The computer calculates mass air-flow, air-fuel ratio, inlet temperature, equivalent horsepower, all of which are combinations of measured variables. Many parameters are converted to standard conditions. Plans for linking to a large computer in a time-sharing network are given.

Clore and Thorley (48L) review the capabilities of microcomputers to increase the adaptability of automatic test instrumentation to changing test conditions. Available hardware and software and choice of systems based on computation speed and input-output feature are outlined. Custom design is also illustrated by a battery powered microcomputer designed to withstand extreme temperature conditions for road testing.

POLLUTION ANALYSIS AND INSTRUMENTATION

Laboratory. Lewis (138L) reviewed the role of analytical chemistry in the automobile industry. Topics covered included: use of IR to inspect new engine oils and monitor exhaust emissions, a radiometric method for engine oil consumption measurement, a laser light-scattering photometer for particle size measurements, a modified Anderson cascade impactor for determining the aerodynamic particle size of exhaust particles from cars operating on leaded and unleaded fuel, new methods for determining Pt loss from exhaust catalysts, HC's, NO, NO₂, and NH₃ in exhaust, rubber in particulate debris from tire wear. Ambrosio and Di Lorenzo (6L) discussed analytical techniques in the study of the evolution of pollutants in rich methane O₂ flames. New methods for the study of acetylene production include thermocouples for flame temperature measurement, optical methods for temperature and soot measurement, GC and MS for HC and combustion product distributions, and emission and absorption spectroscopy for determination of short-lived species. GC analysis of the carcinogenic polycyclic aromatics in soot collected in a special probe showed that the concentrations of pyrene, fluoranthene, anthracene, and phenanthrene increase with increasing flame height above the burner, while benzoperlylenes, coronene and 3,4- and 1,2-benzopyrenes reach a maximum concentration 11-14 mm above the burner.

A number of papers involving GC techniques were published. Fritz and Chang (79L) showed that several Rohm and Haas XAD (macroporous) resins were effective adsorbents for organic and inorganic gases (methane, ethane, NH₃, H₂S, SO₂, and vinyl chloride) and as GC column packings for the separation of gases and relatively volatile organic compounds. The chemical structure of the resin strongly affected the retention of the more polar gases. Separations on an XAD-4 column were: Ne, O₂, Ar, methane, Kr, and Xe, and air, methane, CO₂, ethylene, ethane, H₂S, and SO₂. Datar et al.

(54L) described the use of kaolinite and bentonite as stationary GC phases. Sample gases included O₂, N₂, CO₂, Ar, N₂O, H₂S, and acetylene, with He, Ar, N₂, and O₂ as carrier gases. Elution profiles are given and anomalous detector responses discussed.

Singh and Lillian (208L) developed a pulsed flow coulometric method for the absolute analysis of reactive, electron-absorbing air pollutants which undergo decomposition in a GC column. The method was applied to the determination of sub-ppb mixtures of phosgene in air. Compared to permeation tube standards, an error of less than 15% was achieved when ionization efficiencies were greater than 75%, and this was reduced to 4% at ionization efficiencies greater than 85%.

A number of publications appeared on oil spill identification. A multielement true boiling point GC for monitoring oil pollution and fingerprinting petroleum streams was developed by Davis et al. (55L). The effluent from a programmed temperature GC column was fed into an O-charged quartz combustion tube. CO₂ was detected by nondispersive IR and SO₂ by an FPD. Provisions are made for column backflushing and raising the injection port temperature while introducing O₂. Thus C and S boiling point profiles up to C₄₂ are obtained. Szakasits and Krc (216L) patented this method and apparatus. A sampling device for the recovery of petroleum HC's and fatty acids from aqueous surface films was described by Miget and Kator (157L). A disk of 2-mm thick Teflon was attached to a 4-mm marine Al backing fitted with a hinge, and a wooden pole is attached to the hinge at such an angle that the Teflon face and water surface are parallel. After touching the water surfaces, the disk is washed with CCl₄ to remove organic compounds. The sample obtained is analyzed by GC and MS. This provides a rapid way to identify oil spills. Miller and Calcote (158L) patented an apparatus for the determination of organic C in aqueous solutions. The detector used was an FID equipped with 1 or 2 inlets for the fuel gas and combustion-supporting gas, a sprayer for the sample, an outlet for excess gas, and a collecting electrode near the burner. The fuel gas (H, CO, CS₂, H₂S, or their mixtures) and the combustion-supporting gas (air or O₂) are fed as separate streams or as a mixture. The sample is aspirated into the combustion-supporting gas stream or the mixture or is pumped through a nozzle. The sample is passed through an ion-exchange bed or an electrodialyzer to remove interfering inorganic solutes. Brown et al. (32L) used IR to monitor oil spills in Narragansett Bay. Samples were traced to their source by computer matching of their IR spectra. Salen & Wicander AB (192L) patented a method and device for determining the concentration of small amounts of oil in water. The sample was passed at a known rate through a filter such as polypropene "wool" which absorbs the oil. The amounts of oil absorbed in a given time can be determined by measuring the change in color of the filter or by measuring its change in dielectric constant. The method can be used to determine as little as 10 pm of oil.

Tanimoto and Uehara (220L) used a microwave-cavity spectrometer of the Stark voltage-sweep type to detect acrolein in engine exhaust. By using a preconcentration technique, \approx 5 ppm of acrolein in automobile exhaust gas could be determined. The Cu Stark electrode of the spectrometer was fed with a variable dc voltage and a fixed frequency of \approx 8.9 GHz.

Several papers on particle size determination appeared. Barothy (15L) measured the particle concentration at various sedimentation heights as a function of time in a narrow region by an x-ray beam. The data are presented as a cumulative weight-equivalent sphere diameter relation by an X-Y recorder. Podol'skii and Kalakutskii (181L) described a device for measuring the particle size distribution of powdered materials based on the classification of particles in an electrostatic field into fractions by the differences in charges connected explicitly with their sizes. The total group charges were measured by induction. The measurement was made for 7 fractions of the particle size range 0.4–40 μm . Differences in the reproducibility of the results were \leq 3%. Analysis time was 3 min. Results of measurements of SiC powders were compared with results obtained by sedimentation. Particle size and shape characterization of atmospheric dusts (from flue or stack gases) was accomplished by Byers et al. (35L) using a scanning electron microscope and a computerized technique

to evaluate the images. The particulate matter was collected horizontally on 0.002-in. Al foil or on aluminized Mylar film. A Bendix electrostatic precipitator was used with a standard sampling time of 1 h. The method compared satisfactorily with (1) counting by eye and (2) computer processing of scanning electron microscope images based on the binary mapping system. Only particles ($>0.1 \mu\text{m}$) of nonvolatile matter could be examined.

Process. Several papers and publications reviewed pollution analysis instrumentation and control. Papers were given at an ASTM meeting in Boulder, Colo., in August 1973 on instrumentation for monitoring air quality and reported in the *ASTM Technical Publication*, No. 555 (1974). Subjects covered were continuous monitoring of HC's, performance of HC monitoring instruments, plumbing problems, and methods to obtain reliable service from HC analyzers. Moriya (161L) reviewed natural resources vs. environmental protection, pollutants and their analyses, and analyzers for pollution control. Hood (102L) surveyed new developments in equipment and sampling techniques for automatic analysis of product and effluent streams. Some instruments discussed were: Pye Unicam's on-line TOD meter, used on heat exchanged water for hydrocarbon contamination before boiler return, Westinghouse's direct flue gas oxygen analyzer based on the Hagan fuel cell principle fitted with central loops for various combustion processes, Feedback Instruments Ltd.'s IR analyzer for monitoring CO₂, and Lee Engineering's Sonic Solution Monitor for on-line sound velocity measurement with $\pm 2\%$ accuracy in measuring concentration of 90–100% sulfuric acid.

Klein (124L) surveyed the latest on-line gas and liquid analyzers offered for monitoring O₂, CO, HC's, H₂S, S, particulates, flue gas O₂, total carbon, chlorine, trace elements, and viscosity. Condron and Puzniak (50L) discussed the application of on-line instrumentation for control of water quality in the petroleum industry.

Several papers have dealt with the detection and analysis of vinyl chloride in industrial and ambient air. Vinyl chloride can be detected (110L) to levels lower than 1 ppm with Applied Automations Inc.'s new GC system. Other features include permanent hard copies and visual or audio alarms at predetermined levels. Vinyl chloride was successfully monitored (41L) with a computer/IR system at two PVC plants. The system automatically determines employee exposure through statistically sampling and time-motion studies, thus eliminating the need for direct personal sampling. The plants were able to reduce emissions of vinyl chloride considerably.

In England (42L), vinyl chloride exposure concentrations were reduced from 500 ppm before 1972 to 200 ppm in 1972 to 25 ppm in 1974. Instrumentation now used or being installed are GC, IR, and FID. The analyzers are attached to automatic sequential sampling systems, which sample air at up to 24 points at a rate of 1/min. Further development work continues to improve the sensitivity and portability of the monitors.

Robintech, Inc. (40L) uses a computer-based vinyl chloride monitoring system which runs samples every 30 s, giving continuous time-average analysis of ambient air. Alarms automatically sound if the vinyl chloride concentration exceeds set limits.

Golding (85L) described a portable IR analyzer for air monitoring. The single beam system can detect and quantitatively measure any gas with absorption bands in the IR region. A variable-path cell with a maximum path length of over 20 m permits detection of sub-ppm levels for a variety of organic vapors including vinyl chloride, ethylene oxide, etc.

A personal atmospheric gas sampler using the critical orifice concept was developed by Williams et al. (237L) to collect time integrated air samples for up to 8 h in industrial environments. An evacuated chamber of 100 cm³ with a critical orifice is used to collect samples rather than a pump. The sampling interval is dictated by orifice size and volume of the sample container. Samples are analyzed by GC. Results are presented for various HC's, chlorohydrocarbons, vinyl chloride, CO, CO₂, and other vapors.

A NIOSH developed atmospheric sampler (109L) has proved useful in analyzing worker exposure to industrial solvent vapors. The sampler consists of a glass tube containing sections of activated charcoal, polyurethane foam, and glass

wool. The glass tube is affixed to the worker's collar and a sampling pump fitted in the shirt pocket. Pumping rate is 1 L/min or 10 min. In the laboratory, the chemicals on the charcoal are removed with CS₂ and a portion is analyzed by GC.

Denenberg and Kriesel (60L) describe the use of the Series 7000 continuous area monitors which use chemically-imregnated paper tapes through which the work place atmosphere is aspirated at a controlled rate. The intensity of the stain is measured using reflected light giving a signal related to vapor concentration. Monitors are available for phosgene, toluene diisocyanate, chlorine, SO₂, H₂S, NO₂, and vinyl chloride. Similar tapes are available for miniature continuous monitors which differ from the area monitors mainly by being smaller, and tapes are read at the end of an 8-h exposure period. The range and sensitivities are discussed.

Kieffer (120L) developed a probe for the detection of various organic vapors in air such as ketones, acids, alcohols, paraffins, aromatics, esters, and nitrogen compounds. The "electro-nose" probe produces identifying responses for odorous chemical families. The feasibility of using an odor-monitoring instrument to replace odor-sensing panels is suggested. The solubility parameter was used in selecting a polymer for the probe. The design and operation of the electro-probe are discussed.

Dailey (53L) surveyed the current analytical methods for detecting lower explosive-limits (LEL) or threshold-limit-value (TLV) concentration of industrial materials. Instruments covered were detectors for natural gas, H₂, HC's, and solvents based on GC, flame ionization, TC, IR, or catalytic combustion. For workspace poisons, sensors reviewed include catalytic reaction at filaments, beds, and surfaces, optical absorption, ionization effects, and electrochemical and colorimetric principles. Also surveyed were sampling locations, alarm devices, calibration, data handling, and maintenance. A list of manufacturers and legal requirements is given.

Liptak (143L) discussed hydrocarbon detectors to actuate explosion-suppression systems in milliseconds in closed-processes where fast changes can occur. Both UV and IR detectors are suitable detectors. Conventional catalytic combustion sensors to detect flammable vapors are too slow for use as explosion detectors.

A monitor for explosive gases (179L) developed in Great Britain automatically samples the atmosphere at four-minute intervals and selectively determines the concentration of gases such as town gas, natural gas, propane, or butane. Visible and audible alarms give warning of gas buildup to explosive concentrations. False alarms due to nondangerous substance are eliminated. A variable frequency alarm signal can be used to detect increases in concentration and thus is useful for tracing leaks.

Fredericks and Scott (78L) designed a solid-state type combustible detection unit consisting of a small block of sintered oxides of Sn and Zn. The block is maintained at 250 °C by two internal heaters which also act as electrodes for measuring the resistance of the block. The resistance decreases when a combustible gas is present. Sensitivities for several vapors are given.

Arora (9L) discusses a continuous monitor for CO in industrial workplaces. It operates on the basis of the exothermic oxidation of CO in the presence of the catalyst hopcalite. The rise in temperature is correlatable with the CO concentration and the unit has a minimum detection limit of 5 ppm of CO. The monitor can be used with direct reading meters or continuous recorders and can operate alarms.

Correct calibration is a crucial part in the overall accurate operation of ambient air monitors. McKinley (149L) discusses this subject for the new type of monitoring systems for recording concentrations of various organic and inorganic pollutants. Calibration techniques to be used for instruments in

emission control and workplace environment are: coulometric generation, quantitative conversion from stable mixtures, exponential dilution, and constant emission with blending. The advantages and disadvantages of each method are discussed.

Saltzman et al. (193L) described a Beckman 109A total HC analyzer that was modified to continuously monitor unsaturated HC's in air. Metered streams of air are continuously pumped through a parallel arrangement of an empty column and one packed with CrO₃-H₂SO₄ supported on Chromosorb. The effluent from each column was alternately directed every 5 min to a FID. The difference between the two readings gave a measure of the unsaturated HC's in air.

McLean and Holland (150L) developed a portable polarograph for the determination of aldehydes in automotive exhaust and production plant samples. A 2% hydrazine sulfate buffered solution (pH 4) was used to analyze samples containing 7 to 1500 ppm of formaldehyde. Results obtained agreed with colorimetric methods. Application to monitoring aldehydes in gaseous or aqueous streams is described.

A patent (31L) describes a detection system for CO which provides a signal to control the engine-ignition advance and injection times in order to correct the exhaust gas composition. A sampling tube from the exhaust pipe passes the gas into a chamber divided into two sections by a thermoelectric lead telluride element having a cold surface in one section and a hot catalytic surface exposed in the other section. With CO present a Δt is created in the lead telluride element causing a current to be generated which is passed to an analog-to-digital converter for computer processing. The computer can then regulate the ignition sequence.

Gregory et al. (87L) describe an apparatus for the continuous monitoring of oil-in-water using a fluorescence technique. The system consists of a tubular housing through which the water stream falls as a core in an annulus of air. The upper portion of the housing contains windows through which UV is shown and the fluorescence is detected.

Trace oil in boiler feed water is determined by a simple device according to McAlister (146L). Condensate water from a fuel oil preheater flows through an inlet line and strikes a baffle plate dispersing any oil present. After turbulence is reduced by a partition, a sample line set at water level in the outlet tank collects any oil film on the surface. The water sample drains through a beaker which is checked. If oil is present, it appears as a film on the sides of the beaker and the operator sends the condensate to waste.

Sell and Buening (200L) describe a system for continuously taking liquid samples for further analysis. A light-activated control system rotates a table on which flow-through sample cells are placed. The table includes circles of holes at two different radii. Above the circles of holes are 3 lamps and below the circles are 3 photoresistors which are in the motor control circuit. The 1st lamp controls the standstill time for taking a sample from a flow-through cell with an automatic pipet. The 2nd lamp reverses the direction of rotation, and the 3rd stops the return of the table and turns the table in the opposite direction to the position of sampling. The system was used in analyzing waste water streams.

Hubby (104L) devised an apparatus to determine oil in water draining from an offshore drilling rig. Water is led through the deck into a vertical, longitudinal container that forms part of a U-tube. The outlet leg, containing liquid at a constant height, consists of an open vertical pipe reaching almost to the bottom of the container and discharges through the wall above sea level. The inlet leg is the liquid in the container. When liquid of different density, such as oil, enters, its level is higher than that in the outlet leg. A float operates a pneumatic valve that sets off an alarm or shuts off the delivery of liquid to the container.

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