Solid and Gaseous Fuels

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SOLID FUELS

This section covers methods of sampling, analyzing, and testing coal, coke, and coal-derived solids. *Energy Research Abstracts* and *Chemical Abstracts* were used as reference sources. The volume of material available made it necessary to limit the number of publications in the review.

SAMPLING AND PROXIMATE ANALYSIS

Sampling. A sample-preparation procedure used to produce a bank of samples ranging from low-grade lignites to high-rank bituminous coals was described by Benson et al. (*4A*). Sampling methods, both manual and automatic, for milled peat were discussed by Impola and Okkonen (*12A*). Gills et al. (*10A*) described the National Bureau of Standards protocols for sampling, preparation, and homogeneity testing of four standard reference coals with nominal sulfur contents ranging from 0.5% to 4.5%.

Techniques for sampling and analysis of hard coals were surveyed by Blomqvist et al. (*5A*). Honeyball et al. (*11A*) described an alternative method for sample preparation of coal for examination by infrared spectroscopy.

Moisture and Proximate Analysis. Microwave drying of coals was shown by Jacobs (*13A*) to give results comparable to ASTM methods, with little or no coal degradation and shortened drying times. The water content in coal was determined coulometrically by Bao et al. (*2A*). Weber and Praxmarer (*30A*) employed microwave irradiation for the rapid and continuous measurement of water content in raw brown coal. Luppens (*17A*) discussed the application of the equilibrium moisture test to lignites and the use of ash-moisture plots in evaluating equilibrium moisture results. The impulse-neutron γ-scattering method was applied to the simultaneous determination of moisture, ash, and carbon in metallurgical coke by Dombrovski et al. (*6A*). Pavlovic et al. (*23A*) used Fourier transform analysis to evaluate X-ray diffraction data in the determination of rank, volatiles, fixed carbon, ash, vitrinite, and nonvitrinite mineral content of 29 coals ranging in rank from anthracites to subbituminous. Earnest (*8A*) reviewed the use of computerized instrumentation for the rapid proximate and ultimate analysis of coals. An automated multisample analyzer for the proximate analysis of coal and coke was described by Sitek and Walker (*28A*).

Nuclear magnetic resonance (NMR) was used by Riley (*24A*) to measure the moisture and total hydrogen content of 50 coals and by Rytsar et al. (*26A*) to determine water in coal samples rapidly by using a portable analyzer.

Thermogravimetry was used for proximate analysis in the testing of several varieties of coals and cokes by Ottaway (*20A*); in microprocessor-controlled rapid analysis of coals by Earnest and Fyans (*9A*); in analysis of coals, oil shales, and peats by Sadek and Herrell (*27A*), who compared results with those obtained by ASTM methods; and in conjunction with thermomagnetic measurements of pyrite content by Rowe (*25A*).

Ash. Pandey and Prasad (*22A*) applied an X-ray backscattering technique to the rapid determination of ash content of coke. The rapid determination of coal ash content by means of X-ray fluorescence measurements in three different energy ranges was described by Dziunikowski and Stochalski (*7A*). Neutron activation analysis using an isotopic fast neutron source was employed by Janczyszyn and Stochalski (*14A*) to determine the ash content of 72 pit coals. In addition, precision and accuracy were evaluated, and the results were compared with those from fluorescence-scattering methods. Starchik et al. (*29A*) calibrated a radioisotopic ash meter that can be used for on-line analysis of ores and various coal fractions, including tailings. The effect of moisture content in coal upon the intensity of γ-quanta in the neutron-activation γ-radiation method for the determination of ash content was examined by Pak and Starchik (*21A*). Bartnik (*3A*) discussed the analytical methods suitable for determining the ash content of lignites.

Volatile Matter. Conversion of moisture to C_2H_2 and gas chromatographic analysis of reaction products were employed by Jenke and Hannifan (*15A*) to differentiate between the moisture and volatile components of coal. The effects of one-stage and two-stage heating in the determination of volatile matter in Victorian brown coal were studied by Attwood et al. (*1A*). Nandy and Pal (*19A*) developed a preheating technique to avoid the entrainment of coal particles in the single-stage Indian Standards method for determination of volatile matter in coal. Kopp and Harris (*16A*) examined the relationship of initial volatilization temperature (t) and average volatilization rates (k) to coal rank for 38 coals. Plots of both t and k against percent volatile matter or percent fixed carbon show changes in slope at sharp differences in coalification, which are recognized on the basis of changes in optical and chemical character of the macerals.

Merrick (*18A*) developed mathematical models of the evolution of volatile matter from coal.

ULTIMATE ANALYSIS AND SULFUR FORMS

Nadkarni and Pond (*16B*) separated and determined the concentrations of nitrogen, of sulfur, and of halogens in coal using oxygen bomb combustion and ion chromatography. White and Whittingham (*23B*) determined C, H, N, O, and S simultaneously using two Perkin-Elmer Model 240 elemental analyzers and a Leco automatic sulfur titrator and found the accuracy and repeatability of the data similar to those produced by classical methods. Various rapid coal analysis methods for C, H, N, S, halogens, and metals were employed by Nadkarni (*15B*); results were comparable to those from ASTM methods.

Sulfur. Ivashchenko et al. (*8B*) determined sulfur, ash, and pyrite contents of coals and beneficiated coals with a noncrystal X-ray analyzer. Lawrenz and Sitek (*12B*) analyzed coal and coke for sulfur content by a rapid high-temperature combustion analyzer with microprocessor-controlled infrared absorption. Sulfur in fossil fuels was determined by means of an automated combustion/amperometric titration analyzer by Jarmell (*9B*), while Landsberger et al. (*11B*) determined sulfur in coal by proton activation analysis. Substoichiometric isotope dilution was applied to the analysis of sulfur in coal by Downey and Huston (*3B*). Markuszewski (*14B*) reviewed methods for the determination of total sulfur in coal, highlighting advantages and disadvantages, limitations, and difficulties. Total sulfur in coal was determined by X-ray fluorescence spectrometry by Weber et al. (*22B*) using fusion and subsequent preparation of a borax bead and by Badura (*2B*) using dilution with powdered cellulose.

Oxygen. Frigge (*6B*) described a method for direct oxygen determination in coals, using pyrolysis to CO, which is measured in an integrating IR cell. Yoshida et al. (*25B*) determined ether oxygen in coals by carbon-13 cross polarization/magic angle spinning NMR spectrometry. Oxygen contents of coals were determined both by fast neutron activation analysis and by a pyrolysis/coulometry method by Ehmann et al. (*5B*). Results were in agreement, but they noted some differences when results were compared with ASTM and with Given's oxygen-by-difference methods.

Forms of Sulfur. Scanning electron microscopy coupled with energy-dispersive X-ray analysis was used by Straszheim et al. (*20B*) and by Maijgren et al. (*13B*) to determine organic sulfur in raw and chemically desulfurized coals. Vasserman and Gorelov (*21B*) reported on the accelerated determination of sulfur in coal, using dissolution in HNO_3 and H_3PO_4 and determining the concentration of sulfate turbidimetrically.

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The forms and content of sulfur in Assam coals were studied using X-ray photoelectron spectroscopy by Dutta et al. (*4B*). Two sulfur peaks were observed; one peak was absent in desulfurized coal, indicating correspondence to organic sulfur. Furuya et al. (*7B*) measured the oxidation states of sulfur in coal and fly ash using double-crystal high-resolution X-ray fluorescence spectrometry. Electron probe microanalysis for direct determination of organic sulfur in coal was reported by Raymond (*18B*). Jones et al. (*10B*) studied sulfur forms and nitrogen in coal using X-ray photoelectron spectroscopy.

Wu (*24B*) used EDTA for the indirect volumetric determination of pyritic sulfur in coals. Potential errors in the determination of organic and pyritic sulfur in coal, caused by incomplete dissolution of pyrite in HNO_3, were described by Stanton and Renton (*19B*). A rapid method for determination of forms of sulfur in coal, based upon the sequential liberation of each sulfur form as H_2S, was described by Purnell and Doolan (*17B*). Aylmer and Rowe (*1B*) combined thermogravimetric analysis and thermomagnetogravimetric analysis for the proximate analysis of coal and the determination of pyrite in coal by examination of the ash.

CALORIC VALUE

Benson and Schobert (*2C*) determined the heating values of low rank coals by means of pressure differential scanning calorimetry; results generally compared well with the heating values determined by bomb calorimetry. An X-ray fluorescence method was described by Beaupre (*1C*) for the determination of heating value and ash content of coal. An X-ray backscattering procedure for the determination of caloric value, ash, and moisture for 80 brown coals was developed by Chrusciel et al. (*3C*).

Regression analysis of large data banks of coals of all ranks was used to correlate caloric value with ultimate analysis by Mason and Gandhi (*6C*), by Mason and Talwalker (*7C*), and by Francis and Lloyd (*4C*). The theoretical basis and geochemical implications of the calculation of caloric value of coals from ultimate analyses were discussed, and a new equation including a term explicitly related to the enthalpy of decomposition was proposed by Given et al. (*5C*).

STRUCTURAL CHARACTERIZATION

Petrographic Characterization. Finkleman (*16D*) discussed the origin, occurrence, and distribution of inorganic constituents in low-rank coal. Spiro and Kosky (*55D*) constructed space-filling models for coal and examined their extension to coals of various ranks. Studies on the relation of coal properties to structure were reviewed by Ouchi (*42D*). The optimum classification of coals based on elemental analysis and petrographic parameters was presented by Neavel et al. (*40D*). Boulmier et al. (*5D*) reviewed the applications of transmission electron microscopy in the study of coalification and carbonization. The chemistry and characterization of coal macerals were reviewed by Winans and Crelling (*69D*). The application of multivariate statistical methods in coal petrographic studies was reviewed by Volkova and Tarakanova (*63D*).

Recommended procedures for the petrographic microscopic description of bituminous coal were described by Chao et al. (*7D*). Fluorescence microscopic rank studies on liptinites and vitrinites, and comparisons with results of the Rock-Eval pyrolysis, showing a close relation between the temperature of maximum hydrocarbon release and the classical rank parameter, were made by Teichmueller and Durand (*60D*). Ottenjann et al. (*41D*) found correlations between the fluorescence of vitrinites at an incident wavelength of 546 nm and the swelling index and dilation behavior of bituminous coals.

Sumitomo Metal Industries, Ltd. (*59D*), determined the maceral and mineral constituents of coal by automated stepwise scanning with a polarized light-reflecting microscope. Detection of weathering in coal by petrographic, rheologic, and chemical methods was discussed by Marchioni (*33D*). Wert and Hsieh (*65D*) made a transmission electron microscopy study of the minerals in coal. General topography, crystal type, planar spacing, and chemical composition can be determined. Laser Raman spectroscopy was applied by Green et al. (*22D*) to the identification of minerals, the study of the interaction of inorganic additives with coal, and the structural characterization of coals and cokes; results were compared with conventional optical microscopy measurements.

Maylotte et al. (*36D*) applied X-ray computed tomography to the study of the internal structures of coal and the penetration of tracer gas into those structures. The Leitz texture analysis system of automatic image analysis was used by Riepe and Steller (*48D*) for the microscopic determination of rank and maceral-group composition of coal and coal blends.

Roscoe et al. (*49D*) compared the target transformation factor analysis of coal composition data with X-ray diffraction analysis for quantifying concentrations of mineral matter in whole coal. Coal rank and maceral types were characterized by Crelling and Bensley (*10D*) using fluorescence microscopy.

A method for rapid characterization of a wide variety of bituminous coals by factor analysis of the Fourier transform IR spectra was described by Fredericks et al. (*17D*). Good correlations were found between predictions and measured values of reflectance, volatile matter, hydrogen content, fluidity, and Hardgrove grindability index. A new microscopic IR technique was developed by Brenner (*6D*) to carry out functional group analysis of individual macerals in coals, including new procedures for preparing uncontaminated thin sections of coals. Dyrkacz et al. (*15D*) made a Fourier transform IR study of the nature and magnitude of variations exhibited by various density-differentiated macerals from several coals and found that profound variations in organic structures can exist within a maceral group.

Allen and VanderSande (*1D*) analyzed submicrometer mineral matter in a lignite, a bituminous coal, and a semianthracite via scanning transmission electron microscopy. The predominant mineral species in these inclusions differ from those species dominating in mineral particles at larger size ranges. Petrographic variation across polished coal surfaces was mapped by Davis et al. (*12D*) using automated reflectance microscopy. Reactions in pyrite framboids during electron microscopy of pyrite in bituminous coal were observed

by Harris et al. (*25D*), showing that exposure to the focused high-voltage electron beam causes changes in the sample.

Harper et al. (*24D*) correlated pyrolysis mass spectra of Rocky Mountain coals with ultimate, proximate, petrographic, and mineralogical measurements; predictions of rank and calorific value were made. Individual coal macerals were analyzed in situ in a time-of-flight mass spectrometer via laser pyrolysis by Vastola and McGahan (*62D*).

Molecular Characterization. Subjects of review articles included the following: methods of investigation employed to study functional groups and heteroatoms in coal, by Attar and Hendrickson (*2D*); aromaticity in coal, as inferred by chemical reactivity, X-ray diffraction patterns, optical properties, IR spectroscopy, ESR, and NMR, by Gerstein et al. (*20D*); the use of model compounds in coal chemistry, by Collins (*9D*); modern concepts of the molecular structure of coal, by Lazarov (*31D*); the use of NMR in the examination of solid fossil fuels, by Jones (*28D*); methods used in analysis and characterization of coal, by Rentrop (*47D*).

The determination of carboxyl groups in brown coal was reviewed by Rast et al. (*46D*); electron paramagnetic resonance (EPR) applications to fossil fuels, by Strigutskii et al. (*58D*); changes in physicochemical properties of coals during grinding, by Saranchuk et al. (*50D*); applications of Fourier transform IR spectroscopy in fuel science, by Solomon et al. (*54D*); the application of Mössbauer spectroscopy to coal characterization, by Montano (*37D*); the application of Fourier transform IR spectroscopy to the quantitative determination of functional groups in coal, by Painter et al. (*44D*). Applications of thermal analysis to coal and oil shale were reviewed by Warne (*64D*); aspects of the thermodynamic theory of coal, by Dobal (*13D*); nuclear activation (NAA) methods for the analysis of fossil fuels, by Nadkarni (*39D*); analyses of CH_n (n = 0–3) groups in fossil fuel materials, by Steedman (*57D*).

A model of coal structure consistent with data on swelling, based on electron-donor and electron-acceptor characteristics, mass spectrometric analyses, ^1H NMR, and porosity measurements was presented by Marzec and Kisielow (*35D*). Berkowitz (*3D*) discussed some inconsistencies in current concepts of coal chemistry. Dunbar and Wilson (*14D*) measured the oxygen-18/oxygen-16 ratios of some New Zealand peat, lignite, and coal samples to study the formation and chemical origin of coal. Gaines and Page (*19D*) compared fast atom bombardment, secondary ion, and laser microprobe mass spectrometry as techniques for determining coal structure.

Marzec et al. (*34D*) applied proton pulse NMR to the determination of molecular and macromolecular phases in coals. Conventional and dipolar dephasing carbon-13 NMR techniques were applied to the study of structural variations in coals and coal macerals by Wilson et al. (*68D*). Snyder et al. (*53D*) determined hydroxyl groups in coal by Fourier transform IR and carbon-13 NMR spectroscopy.

Havens et al. (*26D*) characterized a series of vitrinites and studied the low-temperature oxidation of coal by magic-angle carbon-13 NMR and Fourier transform IR spectroscopy. Dack et al. (*11D*) used ESR spectroscopy to study free radical involvement in the oxidation of brown coal and found reversible interactions with oxygen and possible formation of a coal–O complex. Johnson and Cooney (*27D*) used infrared spectroscopy to study the effect of a period of 11 months of weathering on the 1600-cm^{-1} band in Australian bituminous coals of different ranks.

Srinivasan and Seehra (*56D*) used ESR spectroscopy to investigate the effects of pyrite and pyrrhotite on free radical formation in coal, and proposed that enhancement in the number of free radicals is due both to the pyrite-to-pyrrhotite conversion and to the presence of pyrrhotite itself. Whole coals were characterized by carbon-13 CP/MAS spectroscopy at high field by Botto and Winans (*4D*). Mössbauer spectroscopy and diffuse reflectance IR Fourier transform spectroscopy were used by Lin et al. (*32D*) to study low-temperature oxidation of minerals in bituminous coal.

The apparent ratio of quaternary to tertiary aromatic carbon atoms in an anthracite coal was determined by Murphy et al. (*38D*) using carbon-13 proton dipolar dephasing NMR and X-ray diffraction. Schoening (*52D*) developed a new structural parameter that is related to the Fourier transform of the X-ray diffractogram. Cheung (*8D*) applied X-ray photoemission to studies of coal structure. The fraction of carbon in polynuclear aromatic rings can be determined by the asymmetry of the C(1s) line.

The relaxation times of carbon-13 and nitrogen-15 nuclei in solvent-refined coals using various decoupling techniques were investigated by Wilson et al. (*66D*). Aromaticities of coals were estimated by Fukazawa et al. (*18D*) from the IR spectra and ^1H NMR spectra, and oxygen contents were determined by elemental analysis. The proton environment of paramagnetic centers in natural and solvent-refined bituminous coal was studied by Schlick et al. (*51D*) using multifrequency ESR and electron spin echo modulation spectrometry. Wilson et al. (*68D*) discussed exploitation of relaxation in cross-polarization NMR spectroscopy of fossil fuels. Packer et al. (*43D*) studied the quantitative aspects of solid-state carbon-13 NMR of coals and found that not all carbons in bituminous coals are observed by the cross-polarization technique, and thus some reported aromaticities appear to be low. Optimal conditions for quantitative analysis of coal and coal-derived solids by cross polarization and magic-angle spinning carbon-13 NMR spectrometry were elucidated by Yoshida et al. (*70D*). Hagaman and Woody (*23D*) made a statistical coal structure analysis by CP/MAS–carbon-13 NMR spectroscopy and quantitatively evaluated aromaticity parameters. Painter et al. (*45D*) discussed the calculation of coal structural parameters from FTIR and ^{13}C NMR spectroscopic data.

Gethner (*21D*) observed hydroxyl hydrogen in coal by semimicro in situ FTIR difference spectroscopy, while hydroxyl groups were determined for a rank range of British coals by Tooke and Grint (*61D*) using Fourier transform IR spectroscopy.

Larsen et al. (*30D*) examined the Sn Mössbauer spectra of coal derivatives to study the spatial distribution of oxygen in coals.

Krichko et al. (*29D*) described the breaking of C–C and C–O bonds and the formation of aliphatic and alicyclic fragments in coking coal in the presence of γ-radiation.

COKING PROCESS AND COKE TESTING

Relationships between coke-carbon textures and coke properties were studied by the British Carbonization Research Association (BCRA) (*4E*) using scanning electron microscopy. Coke strength prediction methods based on petrographic analysis and dilatometry were applied to Australian coals by Roberts (*24E*). Patrick and Wilkinson (*21E*) examined the high-temperature properties of metallurgical coke by drum test and tensile strength measurements. The variability of coke properties within an individual coke oven was studied by Wagner (*30E*) using physical, chemical, and petrological tests and by Nishioka and Yoshida (*18E*), who evaluated porosity, degree of graphitization, and inherent strength. The quality of coke was predicted by the use of Vr-G diagrams by Chen (*5E*), who tested 177 samples of bituminous coals for coking quality by determining strengths and caking and coking properties, and carrying out proximate, ultimate, and petrographic analyses.

Tsai et al. (*28E*) studied the co-carbonization of SRC with 12 American coals by measuring Giesler fluidity. The porosity of anthracite and of the coke formed from it during heat treatment was investigated by Fialkov et al. (*7E*) and related to the distribution of the inorganic components of the coal. Meltzheim (*15E*) evaluated coking degree by measurement of coke electrical resistance and compared results to H and volatile determinations of cokes. Structure in cokes from coals of different rank was examined both by scanning electron microscopy and by optical microscopy by Qian et al. (*22E*). The influence of volatile matter and deposited carbon on coke yields was examined by Chiu and Hong (*6E*).

Filonenko et al. (*8E*) developed a mathematical method for predicting strength (χ) of coke lumps in blast furnaces based on changes in average lump size and mechanical stress buildup in lumps during heating. Vandezande (*29E*) correlated coke microstructure with rank and blend proportions of the parent coal, with the reactivity of the coke, and with the ASTM stability of cokes from single coals. Relationships of coal structures with carbonyl oxygen or phenolic oxygen predominating to the strength of their cokes were examined by Weinberg et al. (*31E*).

Nippon Steel Corporation (*16E*) evaluated the reactivity and powder content of metallurgical coke by using a partial combustion apparatus. Hung and Chiu (*11E*) investigated the effect of moisture content of coal blends on coking time,

coke properties, and heat consumption. Bensaid et al. (*1E*) used optical and transmission electron microscopy to observe the interaction between coals and pitches during the coking process.

Oya et al. (*19E*) carried out structural studies of cokes using optical microscopy and X-ray diffraction. Sakawa et al. (*26E*) made ESR studies of formation of coke texture, finding a relation between concentration of free spins during carbonization and the coke texture. Gray (*10E*) correlated petrologic maceral composition and vitrinoid reflectance with carbonization characteristics to predict how coals will perform in the coking process and to evaluate coke strength. The influence of oxidation upon strength and structure of a needle coke and a coal-extract coke was examined by Ragan and Marsh (*23E*) using optical microscopy and measuring microstrengths. The BCRA (*3E*) correlated the O–C ratio and fluidity of British coals with the strength and reactivity of the corresponding cokes.

A method and device for determining the coking properties of green coal by image processing was developed by Nishikawa et al. (*17E*). Robinson (*25E*) studied the co-carbonization of American coals with commercial additives, measuring coke strengths and measuring ash, sulfur, and solids contents. Kawasaki Steel Corporation (*14E*) related cokability of coal to the oxygen content of vitrinite and exinite as determined by the JIS M8816 method. Ghose and Chaudhuri (*9E*) predicted coke strength from maceral analysis and reflectance measurements on vitrinites. Using computerized microscopy for automatic image analysis, Patrick and Stacey (*20E*) studied the effects and control of coke porosity. The weathering characteristics of coking coals and the accompanying changes in coke strength were studied by Boyapati et al. (*2E*) by measuring the pressure drop of a stream of nitrogen flowing through a coal sample during continuous heating. X-ray and microscopic characterization techniques were used by Jasienko and Gerus-Piasecka (*12E*) to examine the structure of cokes from coking coal vitrites, and by Tsai et al. (*28E*) to correlate microstrength and industrial drum strength indices of metallurgical cokes.

Khan and Jenkins (*13E*) evaluated a high-pressure microdilatometer for characterizing the thermoplastic behavior of a Pittsburgh seam hvA coal.

INORGANIC CONSTITUENTS

Chen et al. (*10F*) studied variations with depth of the concentrations of S, Ca, Ti, Fe, Zn, Br, and Sr in vitrinites, compared results from synchrotron radiation determinations with results from similar determinations using the Heidelberg proton microprobe, and discussed the advantages and disadvantages of the two techniques. Iron increases from top to bottom of the coal bed, while sulfur decreases from top to bottom of the bed. Wormald and Clayton (*43F*) used a radioisotope neutron source to determine the concentration of the major elements in the combustible and mineral fractions of coal by measuring neutron-induced prompt γ-rays. Good agreement with conventional techniques was found in the case of 14 elements. The volatilities of 58 elements in coals during ashing were studied by Doolan et al. (*13F*) using X-ray fluorescence spectroscopy (XRF), neutron activation analysis (NAA), atomic absorption spectroscopy (AAS), inductively coupled plasma spectroscopy (ICP), specific ion electrode potentiometry (ISE), and optical emission spectroscopy (OES). The multielement analysis of coal ash using sequential ICP was discussed by Rice and Bragg (*30F*). The method determines 12 elements in 45 s, and the standard deviation is well within the ASTM limits. Fast neutron activation analysis for alumina, silica, and ash was applied to bulk coal samples by Borsaru and Mathew (*4F*). Chakrabarti (*9F*) reacted the organic chlorine in coal with hydrogen in the presence of a nickel catalyst and NH_3 and determined the resulting NH_4Cl.

X-ray fluorescence spectroscopy was widely used for determining inorganic constituents in coal. Several examples are the following:

Artificial coal standards for XRF studies were prepared from certified geological materials by Wheeler (*40F*). To account for the matrix effects in the direct determination of major, minor, and trace elements in coal by XRF, Farne et al. (*15F*) used a system of equations, while Janke (*18F*) used energy-dispersive X-ray spectrometry with digital computation of absorption factors to minimize interelement effects in the analysis of fused coal ash. Thorne et al. (*35F*) analyzed 84 bituminous coal samples using wavelength dispersive XRF and found that the accuracy obtained was comparable to that for conventional British Standards methods. Kusmierska and Badura (*20F*) applied XRF to the determination of chlorine in finely ground coal, while Urbanski et al. (*37F*) used XRF to determine the Ca, Fe, and ash contents of brown coal. Calcium fluorescence produced by γ-rays from a ^{55}Fe source was used by Pak (*29F*) for the rapid nondestructive monitoring of Ca concentration in coal.

Trace Elements. Valkovic et al. (*38F*) compared three different modes of sample excitation in X-ray emission spectroscopy for trace element analysis of coal and coal ash. Trace elements in coal and fly ash were determined by Suzuki and Hirai (*34F*) using low-energy photon spectrometry. Inductively coupled plasma atomic emission spectrometry was used for multielemental analysis of brown coal and brown coal ash by Marquardt et al. (*24F*) and by Mahanti and Barnes (*23F*) after separation and concentration on a poly(dithiocarbamate) resin. The mobilities of 40 trace elements in coal during char formation at 400–450 °C and 600–700 °C were examined using X-ray diffraction by Cahill and Shiley (*7F*). Kellerman et al. (*19F*) developed methods for the dissolution of coal and the subsequent determination of nonvolatile trace elements by flame AAS, and of As, Se, Bi, and Te by hydride formation.

Atomic absorption spectrometry was employed as the analytical technique in studies by Yamashige et al. (*44F*) to compare acid digestion procedures with and without hydrofluoric acid for the determination of heavy metals in coal fly ash, and by Lindahl (*22F*), who used combustion of coal in an oxygen bomb followed by acid digestion of the combustion residue. Obrusnik and Posta (*28F*) analyzed an NBS fly ash (SRM 1633a) and coal (SRM 1632a) for trace elements using NAA. Excellent agreement was obtained between the authors' data and NBS values for Al, As, Ba, Ca, Eu, Ga, K, Na, Sr, Ti, U, and V. The report also gives the concentrations of Dy, In, La, Sm, and W in the fly ash and Br and Cl in the coal. Nadkarni (*25F*) determined 40 trace and major elements in coal and fly ash using ICP, XRF, and AA spectrometry, ion-selective electrodes, and calorimetry. The concentrations of 42 trace elements in Canadian coal were determined using NAA, plasma emission spectrometry, and flame AAS by Landheer et al. (*21F*). Material balances for 62 elements in coal burned in a power plant were obtained by Welcomer (*39F*) using spark-source mass spectrography. Differential-pulse polarography was used by Somer et al. (*33F*) to determine Fe and trace elements in coal.

Neutron activation analysis was widely used for trace element determinations in coal. Both thermal and epithermal irradiation were used for multielement determination, including some lanthanides, uranium, and thorium, in Brazilian coals by Atalla and Requejo (*1F*); epithermal NAA was used by Bernedo (*2F*) to determine uranium and thorium in Brazilian coals. Chvi (*11F*) studied the distribution of gold and platinum in 65 low-temperature-ashed samples of Kentucky No. 9 coal using NAA with radiochemical separation, while Carpenter and Filby (*8F*) determined the fate and distributions of 35 trace elements in the Solvent-Refined Coal-I and -II processes by NAA in conjunction with fission-track analysis for uranium. Photon activation analysis and NAA were applied to the study of the variability and mobility of sodium in low-rank coal deposits and to multielement measurements in coal and ash materials by Robertson et al. (*32F*). Cahill et al. (*6F*) compared NAA with other analytical methods for the analysis of 35 elements in coal. Agreement with values from XRF analysis for Fe and K was good; agreement with AAS values for Ni content was poor. The NAA values agreed with values for 35 elements in a coal reference standard. Burmistrov and Chekanov (*5F*) developed a method for the determination by NAA of rhenium reserves in coal seams.

Bettinelli (*3F*) determined fluorine in coal and in environmental standard reference materials with a fluoride ion selective electrode, after sample combustion in a high-frequency combustion furnace. Mercury, As, Se, and Mn in coal were determined by Wilkinson et al. (*41F*) using cold-vapor atomic fluorescence spectrometry, AAS, and ICP/AAS. The determination of Ge in coal ash using ICP with an echelle high-resolution atomic emission spectrometer was reported by Nadkarni and Botto (*26F*).

The mode of vanadium occurrence in high-volatile bituminous Kentucky coal was examined by Wong et al. (*42F*) using X-ray absorption spectroscopy. Garbauskas and Wong (*16F*) carried out XRF analysis of trace titanium in coal using mathematical matrix corrections and doped graphite standards.

Graphite furnace atomic absorption spectroscopy (AAS) for the analysis of arsenic in coal was used by Ebdon and Pearce (*14F*) with direct atomization of a coal slurry and by Riley (*31F*) to study significant reactions of aluminum, magnesium, and fluorine during the analysis of arsenic. Nakashima et al. (*27F*) compared coal digestion methods for the AAS determination of cadmium in coal.

Coal and coke samples were analyzed for trace mercury by Uchikawa et al. (*36F*) using flameless atomic absorption spectrometry with a simultaneous background detection system that revealed the background molecular absorption. A high-temperature combustion technique combined with cold-vapor atomic absorption spectrometry for the determination of mercury in coal and coal products was utilized by Doolan (*12F*) in conjuction with a glass vapor-generation apparatus designed to produce nontransient signals suitable for either analog or digital recording devices. The Jerome gold-film procedure was used by Heunisch (*17F*) to determine trace mercury in coal, coal slags, and fly ash.

ON-LINE ANALYSIS

The on-line determination of the ash content of coal using a "SIROASH" gauge based on the transmission of both low- and high-energy γ-rays was described by Fookes et al. (*4G*). LeVert and Managan (*9G*) used a n/n-type high-purity germanium detector for prompt neutron-activated γ rays to determine major elements and sodium and chlorine in coal. The ash content of a tailings stream was measured by Boyce (*1G*) using two radioisotope-detector systems, a ^{137}Cs d transmission gauge and a Compton backscatter X-ray gauge with ^{109}Cd. The performance of a Sulfurmeter in measuring continuously the sulfur content of coal entering a power station was described by Ruby et al. (*11G*). By measuring the intensity of scattered γ-rays emitted by a ^{241}Am source, Onishchenko et al. (*10G*) monitored the ash and sulfur content of granulated coal on conveyor belts and found that under optimum conditions, the relative errors of the ash and S determinations were lower than the errors involved in sampling for chemical analysis. A prompt γ-neutron-activation method with a ^{252}Cf source, suitable for on-line analysis, was used to determine major elements, Na, K, and Ti in coal by Herzog and Wilde (*6G*) with results in good agreement with chemical analyses. The performance of an on-line nuclear activation analyzer for determining sulfur in low- and high-sulfur coal blends at a power plant was examined by Tassicker et al. (*12G*). Simultaneous utilization of neutrons and γ-rays from californium-252 was used by Tominaga et al. (*13G*) to measure moisture and density of coke in the iron-making process. King et al. (*7G*) described the on-stream measurement by NMR and ESR of the moisture, volatile, C, and ash contents of pulverized coals transported pneumatically in a $^3/_8$-in. pipe.

Klatt (*8G*) reviewed the use of automated on-line analytical instruments in the coal preparation industry, while Clayton and Wormald (*2G*) reviewed neutron interaction techniques and the use of low-energy X-rays and γ-rays for coal analysis, in applications that range from borehole logging to on-line analysis in coal preparation plants and power stations. The potential of nuclear methods for development of novel on-line sensors for coal analysis was discussed by Herzenberg (*5G*). The state-of-the-art of on-line coal analysis instrumentation was reviewed by Cooper (*3G*).

MISCELLANEOUS

Perry et al. (*7H*) used matrix isolation fluorescence spectrometry to determine polycyclic aromatic hydrocarbons in unfractionated solid solvent-refined coal. Coal and coal ash surfaces were characterized semiquantitatively for major elements using an ESCA technique by Brown et al. (*1H*). The surface compositions of 19 coals ranging from anthracite to brown coal were measured for O, S, N, Cl, Si, Al, and Fe by X-ray photoelectron spectroscopy and compared with the results of bulk analysis by Perry and Grint (*6H*); oxidation and carbonization of a bituminous coal were also carried out. Radiation spectrometry on 3-kg samples was used by Salmon et al. (*9H*) to examine the radioactivity of 20 United Kingdom coals. Nonaqueous titration of a bituminous and a subbituminous coal and of coal-derived liquids was carried out by Dutta and Holland (*3H*) to determine carboxylic and phenolic groups. Rhoads et al. (*8H*) investigated the low-temperature oxidation of coal by using curve-resolving methods to distinguish between various C–O functional groups by Fourier transform infrared spectroscopy and by measuring changes in thermoplastic properties with a Gieseler plastometer.

Parallel microcalorimeter–gas chromatographs were used to study the low-temperature chemisorption of oxygen in coals without desorption of the oxidation products by Koshovskii et al. (*4H*), who found that specific surface areas and particle size distributions were similar for Donets coals of different ranks. Surface and bulk low-temperature air oxidation of coals was monitored by Fourier transform IR spectroscopy with a photoacoustic detector and software-controlled spectra subtraction by Donini et al. (*2H*).

The porosity and pore-size distribution of brown coal were determined by Wagenfeld et al. (*14H*) using both X-ray small-angle scattering (SAX) and CO_2 adsorption, while Setek et al. (*12H*) determined the micropore volume and surface area of both wet and dry brown coal by SAX, and compared the technique with the gas adsorption method. Schmidt (*10H*) found that coal rank is the main factor determining the form of the SAX curves for 15 coals of the PSOC series from the Penn State Coal Sample Bank and Data Base, and compared specific surfaces calculated from the scattering data with specific surfaces obtained by N adsorption at low temperatures and by CO_2 at room temperature. Tricker et al. (*13H*) obtained, from small-angle neutron scattering data (SANS), the pore size distribution functions of coals of different rank, which were quantitatively consistent with data obtained from adsorption measurements, thus providing a firm foundation for using SANS to determine coal porosity under conditions where conventional gas adsorption techniques are inappropriate.

Lesko et al. (*5H*) applied differential thermal analysis (DTA) to the study of coal during thermal degradation. Thermogravimetric analysis of coals was compared with DTA for calculation of activation energy, order of reaction, and preexponential factor by Serageldin and Pan (*11H*).

STANDARD METHODS

The development and standardization of test procedures for coal and coke are coordinated by ASTM (*1I*) through the D5 committee. Standards that were adopted during the period covered by this review are the following: D 4182-82, Evaluation of Laboratories Doing ASTM Sampling of Coal and Coke; D 4328-84, Major and Minor Elements in Coal and Coke Ash by X-Ray Fluorescence; and D 4239-83, Sulfur Analysis by High-Temperature Tube Furnace Combustion Methods.

Standards that were revised are the following: D 720-83, Free-Swelling Index of Coal; D 2492-84, Forms of Sulfur in Coal; D 1756-84, Carbon Dioxide in Coal; D 3177-84, Total Sulfur in the Analysis Sample of Coal and Coke; D 3179-84, Nitrogen in the Analysis Sample of Coal and Coke; D 3180, Method for Calculating from As-Determined to Different Bases; D 2795-84, Analysis of Coal and Coke Ash; D 3178-84, Carbon and Hydrogen in the Analysis Sample of Coal and Coke; D 3176-84, Ultimate Analysis of Coal and Coke; D 4208-83, Chlorine by Oxygen Bomb/Ion Selective Electrode; D 2796-82, Definition of Terms Used for the Megascopic Description of Coal and Coke Beds and Microscopical Description and Analysis of Coal; D 3175-82, Volatile Matter in the Analysis Sample of Coal and Coke; D 3286-82, Gross Calorific Value of Solid Fuel by the Isothermal-Jacket Bomb Calorimeter; D 3302-82, Total Moisture in Coal; and D 197-82, Sampling and Fineness Test of Pulverized Coal.

GASEOUS FUELS

This review surveys publications concerned with methods for the chemical, physical, and instrumental analyses of gaseous fuels and related materials. Articles of significance appearing in foreign journals and the patent literature that were not available at the time of the last review are also included. *Chemical Abstracts* and *Energy Research Abstracts* were used extensively as reference sources. Some selectivity was necessary in order to include the most pertinent publications in preparing this review.

GENERAL REVIEWS

Analytical methods for natural gas, refinery gas, and manufactured gases were included in a review by Terrell (*6J*). The instrumentation and applications for gas chromatography in natural gas analysis were reviewed by Huber (*4J*). Equipment, techniques, and the operation of gas chromatography for natural gas analysis were reviewed by Toole (*7J*) and by Hefley (*3J*). Gas chromatography fundamentals in natural gas analysis were reviewed by Durbin (*2J*) and by Tramel (*8J*).

The technical basis for determining the heating value of natural gas was reviewed by Sharples and Pannill (*9J*) in relation to controversy over a U.S. Court of Appeals ruling that vacated Order 93 of the Federal Energy Regulatory Commission. Included was a discussion of the effects of water and other nonhydrocarbon components on heating-value calculations. Kilmer (*5J*) reviewed natural gas custody transfer methods for volume and energy measurements, including pipeline balances, and calculations for the conversion of volume to energy measurement.

Methods used in the calculation of natural gas properties were reviewed by Starling (*10J, 11J*). Past, present, and likely future calculation methods were discussed, with special emphasis on the calculation of the compressibility factor. Caffey (*1J*) reviewed techniques of LPG measurements.

GAS CHROMATOGRAPHY

Gas chromatography continues to be a widely used technique for the analysis of gaseous fuels and related materials. The field of gas chromatography was reviewed extensively by Karasek et al. (*10K*) in 1984. This review covers only those publications directly related to gaseous fuels.

Osjord and Malthe-Soerenssen (*13K*) separated N_2, O_2, and about 60 hydrocarbons (C_1–C_{10}) in natural gas using a combination of a split injection, temperature-programmed (from −30 °C) capillary column for the hydrocarbons and isothermal (50 °C) packed columns for the permanent gases. Columns were molecular sieve 13X, Porapak R, and Chrompack Sil 5 (fused silica). The need for repeatable sample size and frequent recalibrations was eliminated by using relative weight response factors for quantification. Two precut columns in parallel were used at Kitakyushu L.N.G. K.K. (*2K*) to separate C_{2+} compounds from air and methane prior to the gas chromatographic analysis of natural gas, thus eliminating peak tailing and "ghost" peak problems. Tsyguleva (*19K*) analyzed natural and industrial gases with two columns where hot wire detectors acted alternately as detecting and comparing units as the gas was switched between columns. Hydrocarbons (C_{2+}) and CO_2 were determined on a 4-m column filled with 30% triethylene glycol butyrate; and O_2, N_2, and CH_4 were determined using a 2-m column filled with CaA zeolite. The rapid determination of N_2, CO, and C_{1-7} alkanes in natural gas using a single Poropak N column was reported by Skrbic et al. (*16K*).

Sandra et al. (*15K*) discussed the theory of thick-film capillary columns and described an application of this method to the quantification of aromatic compounds and tetrahydrothiophene in natural gas. A single-run natural gas method using a thick-film methylsilicone capillary column for the determination of $C_{>2}$ hydrocarbons and a combination of Porapak and molecular sieve columns for inert gases and C_{1-2} hydrocarbons was given by Huber and Obbens (*7K*). Chen et al. (*4K*) prepared a high-efficiency, porous-layer, open-tubular column coated with a mixture of 13X and 10X molecular sieves and analyzed natural gas within 12-min runs. Column capacity mode injections without splitting were possible.

Hackett and Gibbon (*5K*) reviewed the automation of on-line gas chromatographs for process monitoring and analysis, especially in relation to coal gasification. A fully automated hardware system for the 4-min, on-line analysis of hydrocarbons up to C_8 and inert gases in natural gas was reported by Huber (*8K*). Palo and Walters (*14K*) described a fully automated multicolumn, multivalve system for the analysis of natural gas using a modular design and based on a double series bypass arrangement.

The resolution of sampling and sample-delivery problems in natural gas gathering and transmission was reported by Belyeu (*3K*) through operation of mobile chromatographic laboratories and sample probes on all gas sources. Terry and Hawker (*18K*) described an experimental, portable, automatic natural gas analyzer using miniaturized gas chromatographs made of micromachined silicon wafers. Analysis time was 1 min, and software was developed to calculate Btu and specific gravity. An apparatus, of simple construction, to improve column temperature control in the analysis of natural gas was described by Hu (*6K*); it employs a liquid CO_2 cooler with a solenoid valve, and a heater with an integrated electronic circuit. Gasukuro Kogyo K.K. (*1K*) reported a simple and inexpensive system for fuel and city gases that employs a single oven and a six- or eight-port sampling valve.

Problems in the gas chromatographic analysis of natural gas liquids were discussed by Winfrey (*20K, 21K*). Mooney et al. (*12K*) analyzed C_{1-5} gases in LPG, including dienes and acetylenes, with a capillary column modified with a capillary injection port and valve. The separation of CH_4, C_2H_6, *n*-propane, *n*-butane, and isobutane was reported by Takizawa (*17K*) using a molecular sieve packed column of 1000 × 4 mm internal diameter. The mechanism of adsorption was discussed. Kriegler and Zsiga (*11K*) used an OV-17 silicone column to determine naphthalene in city gas. Hut et al. (*9K*) determined the stable isotope ratios for methane and nitrogen in natural gas by chromatographic separation in milliliter quantities followed by mass spectrometry.

SULFUR COMPOUNDS

The continuous monitoring of hydrogen sulfide and total sulfur in LPG was reviewed by Wengrovitz (*11L*). Richter (*9L*) reported that Radas 1G process analyzers used to determine hydrogen sulfide have operated efficiently in control loops at a large natural-gas-sweetening plant with an uptime over several months of better than 98%. Moen (*7L, 8L*) discussed the application to natural gas, LPG, and reformed gas of a sulfur-monitoring method that employs the electrolytic generation of bromine as a titrant and a coulometric bromine sensing electrode system. A 5% $CdCl_2$ solution saturated with Na_2SO_4 was used by Kushnir and Vivchar (*5L*) for the selective absorption of hydrogen sulfide in the analysis of fuel gases containing carbon dioxide.

A portable digital instrument for the potentiometric titration of volatile thiols in natural gas employing a reverse titration method with a sulfide-selective indicator electrode was reported by Garai et al. (*1L, 2L*). Grigar et al. (*4L*) gave a procedure for determining organic sulfur in fuel gases through combustion of the gas and the coulometric determination of SO_2. The gas chromatographic determination of mercaptans and thioethers in natural gas, where H_2S interference is prevented by using a filter made of activated silica gel impregnated with $CdSO_4$ and H_3BO_3, was reported by Yang (*12L*). Zhurov and Kuznetsov (*13L*) determined mercaptans in natural gas and condensates by concentrating the mercaptans in a solution of alkali, reacting with ammoniacal $AgNO_3$, and decomposing with HCl, followed by gas chromatographic analysis.

The gas chromatographic determination of sulfur compounds in natural gas using flame photometric detection was described by Macak et al. (*6L*). Villalobos et al. (*10L*) used a dual flame photometric detector system to analyze sulfur compounds, such as H_2S, SO_2, COS, CS_2, and lower mercaptans, in natural gas and coal gasification product gas. Gawlik (*3L*) discussed several gas chromatographic methods for determining sulfur compounds in natural gas, as well as a flameless oxidation, conductometric method for determining the total concentration of sulfur compounds.

WATER VAPOR

Methods and apparatus for determining the water vapor content and hydrocarbon dew point of natural gas were reviewed by Dodds (*3M*) and by Murphy (*7M*). Review articles by Kwiatkowski (*4M, 5M*) described titrators for determining moisture in natural gas. The continuous monitoring of the water dew point of natural gas was reviewed by Wilby (*9M*).

Bozeman (*2M*) described an automatic Karl Fischer titrator for the continuous monitoring of moisture in natural gas streams. Amirov and Zakharova (*1M*) determined water in liquefied natural gas by absorption on P_2O_5, followed by electrolysis using a unit coupled to a gas chromatograph for the complete analysis of the sample.

A table and graph of natural gas dew points vs. pressure from 1 to 7 MPa was given by Macak et al. (*6M*). Scheloske

et al. (*8M*) published experimental dew points and enthalpies for sweet and sour natural gases containing various amounts of moisture.

CONDENSATES

Pomykala (*5N*) described the chromatographic analysis of natural gas condensates and the application of the *n*-alkane to isoalkane ratio to natural gas prospecting. The rapid determination of hydrocarbon groups in natural gas condensates using a high-performance liquid chromatography system with both UV and dielectric constant detectors was reported by Ermol'ev et al. (*2N*). Pulz et al. (*6N*) described the analysis and occurrence of carboxylic acids in water condensates from natural gas stored underground.

Methods for calculating the viscosity of natural gas condensates were discussed by Filippov et al. (*3N*) and by Magomadov and Vyrodov (*4N*). Razamat et al. (*7N*) was able to estimate the condensate content (C_{5+}) of formation gas with 10% accuracy. The molecular weight of a mixture of natural gas and condensates was calculated by Abbasov (*1N*) from gas and condensate flow rates.

CALORIMETRY

Methods for determining the calorific value of natural gas were reviewed by Van Rossum (*11P*) and Sypolt (*9P*), and combustion instruments were reviewed by Stern (*7P*). The Yamatake-Honeywell Co., Ltd. (*1P*), measured the heating value of natural and fuel gas using catalytic combustion in a cell that includes an electrothermal wire laminated with sintered metal oxides as a temperature sensor. The Wobbe index of combustible gases was determined by Szonntagh et al. (*10P*) in a controlled combustion system where a ZrO_2 detector measures the oxygen content of the combustion gases and regulates the gas–air ratio. Acoustic sensors that monitor the Btu content of natural gas in a pipeline were described by Watson and White (*12P*).

Huber (*4P*) reviewed 10 American Society for Testing and Materials and International Standards Organization gas chromatographic methods for calculating the calorific values, Wobbe indexes, and densities of natural gas. Methods for the on-line utilization of a gas chromatograph in determining the calorific value of natural gas were described by Sund (*8P*) and by Haas (*3P*).

Kilmer (*5P*) gave methods for changing natural gas custody transfer by volume to custody transfer by heat content. Williams (*13P*) described onboard measurement techniques for calculating LNG and LPG heating values for custody transfer. Deriving heating values of natural gas from its components was discussed by Armstrong and Jobe (*2P*). Skrbic et al. (*6P*) gave a nomograph for determining the calorific value of natural gases from compressibility factors.

DENSITY AND SPECIFIC GRAVITY

Determining LNG densities for custody transfer was reviewed by McCarty (*6Q*). Tramel (*11Q*) described the design and specifications for a balance-type recording gravimeter for natural gas and discussed the principles of operation. Siegwarth and LaBrecque (*10Q*) evaluated commercial densimeters for LNG against a reference gravimetric desimeter and found calibration errors about 10 times larger than their precisions.

Methods for determining the ideal and real specific gravity of natural gas based on gas composition were given by Hutchins (*5Q*). On-line gas mass density measurement was discussed by November (*8Q*) as a possible improvement for flow-metering system accuracy. Caffey (*2Q*) and Beaty (*1Q*) reviewed the calculation of mass measurements for natural gas liquids from volume and density measurements. Calculation of LNG densities from composition data was discussed by McCarty (*7Q*), Haynes (*3Q*), and Roncier et al. (*9Q*), while calculation from new experimental dielectric constant data was presented by Haynes and McCarty (*4Q*).

SAMPLING

Natural gas sampling by continuous on-line, weighted sample, and spot-sampling modes was described by Howard (*2R*). Drake (*1R*) reviewed the sampling of mistlike natural gas streams, where heating-value determination requires special measures be taken. Methods for sampling flowing liquefied natural gas streams were reviewed by Trotta (*6R*) and applied to ship-to-truck transfer.

The deposition of aerosol particulates in sampling tubes and sampling lines during coal gasification was reviewed by Stenger and Bajura (*5R*). Mahalingam (*3R*, *4R*) described experiments and mathematical models for particulate sampling in both straight-tube and continuous-stirred-tank coal gasification reactors.

MISCELLANEOUS

Natural gas metering techniques in relation to gas properties were reviewed by Tefankjian (*32S*) and Balogh et al. (*5S*). Hall et al. (*15S*) recommended the use of mass-flow-rate to volume-flow-rate measurements for natural gas. Mass measurement on an 80%:20% ethane:propane stream was discussed by McCane (*20S*). Gaebler (*11S*, *12S*) determined the effect of the dynamic viscosities' pressure and temperature dependence and of the isentropic exponent on natural and city gas volume flow rate measurement by differential-pressure gas meters.

The deviation from ideal gas behavior ("real-gas factors") of methane and five natural gases was determined by Achtermann et al. (*1S*) from their refractive indexes. Oltean et al. (*24S*) and Meyer and Soles (*22S*) evaluated methods for calculating the compressibility of natural gases rich in carbon dioxide in relation to experimentally determined values. Use of the more accurate supercompressibility factor for natural gas engineering applications was recommended by Starling et al. (*30S*) and by Ghannudi et al. (*13S*). Programs for calculating natural gas compressibility were given by Oltean et al. (*25S*) and by DeGrisogono (*9S*).

Coal gasifier instrumentation needs were discussed by McDaniel (*21S*), while on-line instrumentation to monitor gaseous species was reported by Page and Rhodes (*26S*). Reed (*27S*) described some of the problems encountered when applying thermocouple thermometry to coal gasification. In situ gas analysis in coal gasification by way of Fourier transform IR spectroscopy was reported by Solomon and Hamblen (*29S*). Lamey et al. (*18S*) identified typical higher molecular weight compounds in coal gasifier streams by way of capillary gas chromatography–mass spectrometry and headspace gas chromatography. Lamey et al. (*19S*) described a procedure and results for the analysis of condensable tars in the product steam of a coal gasifier that employed solvent extraction followed by GC/MS, UV, and IR.

Only a few examples of the many recent patents and articles describing combustible gas detectors and sensors can be given here. A catalytic-combustion thermometer-type gas detector was reported by the New Cosmos Electric Co., Ltd. (*2S*). Ikegami et al. (*16S*) and the Riken Keiki Fine Instrument Co., Ltd. (*4S*), described semiconductor-type combustible gas detectors. The characteristics of SnO_2 based combustible gas sensors were evaluated by Clifford and Tuma (*7S*, *8S*). Bingham et al. (*6S*) described a portable, fast-response IR radiometer for detection of liquefied natural gas spills. The optical remote sensing of combustible gases were described by Inaba (*17S*), and remote sensing with laser beam scanning, by the Osaka Gas Co., Ltd. (*3S*).

Voogd et al. (*33S*) reported the fast and accurate quantitative monitoring of seven main components in natural gas using mass spectrometry and a new calibration procedure. Electrical properties, such as the dielectric breakdown voltage, of liquefied natural gas and liquid methane were measured by Takaoka et al. (*31S*) in relation to the hazards of static electricity. Finney and Lindsay (*10S*) described a data processing system used for storage, calculation, and reporting of the compounds analyzed in over 11 000 natural gas samples a year. The compositions of 224 U.S. natural gases were tabulated by Miller and Hertweck (*23S*) with special attention to helium content. A portable analyzer for helium in gas or water was described by Gupta (*14S*) for use in geochemical prospecting. Quadrupole mass spectrometry was used by Sano et al. (*28S*) in analyzing for rare gases in natural gas.

STANDARDS

Monkres (*5T*) reviewed standard measuring methods for the natural gas industry with emphasis on ethane-rich streams. Standard gas chromatography methods for the analysis of demethanized natural gas streams were discussed by McCann (*4T*). Hines et al. (*2T*) reported on a 21-laboratory testing program to develop a statement of precision for the gas

chromatographic analysis of natural gas as defined in the Gas Processors Association (GPA) Bulletin 2261-1972. This report also included a 17-laboratory round-robin test of Btu measurements with field calorimeters using the same gases as the gas chromatography study.

The American Society for Testing and Materials (ASTM) and the International Standards Organization (ISO) develop and standardize procedures for the analysis of gaseous fuels. Recent ISO standards (*3T*) include the following natural gas analyses: 6326/2 (Part 2), Gas Chromatographic Method Using an Electrochemical Detector for the Determination of Odoriferous Sulphur Compounds; 6327, Determination of the Water Dew Point of Natural Gas – Cooled Surface Condensation Hygrometers; 6568, Simple Analysis by Gas Chromatography; 6569, Rapid Analysis by Gas Chromatography; 6570 (Parts 1, 2, and 3), Determination of Potential Hydrocarbon Liquid Content; 6974, Determination of Hydrogen, Inert Gases, and Hydrocarbons up to C_8 – Gas Chromatographic Method; 6976, Calculation of Calorific Value, Density, and Relative Density; 6977, Determination of Water and Methanol Contents – Gas Chromatographic Method. Standards for the preparation of calibration gas mixtures, along with other related standards, are also available from the ISO.

The ASTM standards (*1T*) that have been adopted or revised during this review period include the following: D1071-83, Measurement of Gaseous Fuel Samples; D1265-82, Sampling Liquefied Petroleum (LP) Gases; D1826-83, Calorific Value of Gases in Natural Gas Range by Continuous Recording Calorimeter; D1835-82, Specification for Liquefied Petroleum (LP) Gases; D2163-82, Analysis of Liquefied Petroleum (LP) Gases and Propylene Concentrates by Gas Chromatography; D2650-83, Chemical Composition of Gases by Mass Spectrometry; D3984-82, Ethane Thermophysical Property Tables; D4084-82, Analysis of Hydrogen Sulfide in Gaseous Fuels (Lead Acetate Reaction Rate Method); D4362-84, Propane Thermophysical Property Tables.

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Food

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This review covers the literature for approximately the period from October 1982, the end of the interval of our last report (*7P*), to October 1984. We continue our practice of citing domestic and the more widely circulated foreign journals in preference to less accessible publications when work of a similar nature is reported.

New editions of methods manuals which are available are the eighth edition (1983) of "American Association of Cereal

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