

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 covered during the period of Oct 1984 through Sept 30, 1986. *Energy Research Abstracts* and *Chemical Abstracts* were used as the reference sources. In most categories the volume of material available made it necessary to limit the number of publications in the review.

SAMPLING AND PROXIMATE ANALYSIS

Sampling. Cox's (6A) concern for a coal sample bank that would eliminate the lack of uniformity in the coal samples led to the development of a bank of uniform coal samples from the Premium Coal Sample Program at Argonne National Laboratory, as discussed by Vorres and Janikowski (26A). Ring and Hansen (22A) certified three South African coals as South African reference materials (SARMs). The materials are designated as SARM18, SARM19, and SARM20. The initial sampling of coal was in a lot of 150 kg; these lots were then separated into smaller lots for shipment to 28 laboratories in 10 countries for a variety of analyses. A study of the quality assurance was conducted on various fossil fuels (e.g., coal, coke, fly ash, fuel oils, motor fuels, and shale oils) in relation to standardization of methods and availability of standards by Nadkarni et al. (19A).

Methods and models for coal sampling, sample preparation, and analysis were surveyed by Gleit et al. (12A) and Hissink (14A), and a model was presented for predicting emissions, taking into consideration the sulfur content of the coal. Lyman (17A) gave a simplified explanation and illustration of some basic aspects of coal sampling. Kelly (16A) discussed the mechanical coal-sampling systems and the various experimental errors involved in their use.

Moisture and Proximate Analysis. Gethner (9A) found that vacuum drying at 100 °C may alter the chemical composition of a coal. Thus overall spectroscopic and chemical changes observed in moderate temperature reaction studies may depend upon sample pretreatment, drying and storage. Microwave drying of coals was shown by Jacobs (15A) to be quick and safe and to give moisture-content values comparable to the standard methods. Summer and Morrow (25A) evaluated the use of a microwave-drying technique for determining the total moisture in coal ground to 8 mesh. Drying time was 20 min, and results were close to those obtained by using the standard methods, but with a wider variability. For determining water in lignite, Patzman (20A) developed a quick lignite-drying technique and an automated moisture determination by the Karl Fischer method. Sato et al. (24A) developed an apparatus to measure the moisture content of coke used in steel making. Miyauchi (18A) used infrared (IR) absorption to measure moisture in coal. Weber and Praxmarer (33A) investigated a new method for determining moisture in brown coal, dry brown coal, and brown-coal briquets. It is based on the interaction between moisture and high-frequency electromagnetic radiation. Rapid proximate and ultimate analyses of coals were described by Glass and Roberts (11A) and Pavlovic and Renton (21A). Warrington (29A) discussed a thermobalance with a microfurnace to obtain proximate analysis in 15 min on coal and coke samples. Aylmer and Rowe (2A) and Warne (28A) analyzed coals simultaneously for pyrite and proximate values.

Ash. A device for automatic ash determination in lignite was described by Weber et al. (32A). Arai et al. (2A) developed an analyzer (using an infrared (IR) furnace) to determine ash and sulfur. Foreman (7A) modified the ASTM method by drying in a microwave oven and ashing in a fused-quartz crucible in a muffle furnace. The total time for analysis was 35 min. Buchvarov et al. (5A) developed an automatic ash

determination apparatus based on reflected β particles. The absolute error of measurements was discussed for laboratory and industrial conditions. Wagner et al. (27A) used X-ray fluorescence (XRF) and low-energy X-ray scattering (XRS) for the determination of ash in brown coal. Watt et al. (31A) employed X- and γ -ray techniques to determine the ash content of coal. Borushko and Starchik (4A) and Ryashchikov (23A) discuss the accuracy of ash determinations by employing neutron activation analysis (NAA). The application of an X-ray backscattering method for the determination of ash in coal and the radioactivity of the resulting ashes and slag are discussed by Garzon et al. (8A) and Grabov et al. (13A).

Volatile Matter. Watanuki et al. (30A) studied the accuracy of thermogravimetric analysis (TGA) for determining the volatile matter content in coals; the values obtained from TGA were lower than those from Japanese standard methods. Gieskieng (10A) described an automatic analyzer for the determination of volatile matter in coal and coke. Warrington (29A) found that the thermobalance could be used to obtain the "burning profiles" and the "volatile release profiles" of coal and coke. Using photoacoustic Fourier transform IR spectroscopy (PA-FT-IR), Zerlia (34A) examined both high- and low-rank raw coals for volatile matter.

ULTIMATE ANALYSIS AND SULFUR FORMS

Nadkarni et al. (13B) used the LECO CHN-600 analyzer for rapid and simultaneous determination of carbon (C), hydrogen (H), and nitrogen (N) in coal solids and liquid products. They obtained good precision (± 0.1 -2%) and accuracy (± 0.5 -2%). Dean and Chiu (4B) used ion chromatography for rapid and precise analysis of sulfur (S potential), nitrogen (N), chlorine (Cl), and fluorine (F) in low-sulfur-content coals. Gent and Wilson (6B) determined S and Cl in coal using the Eschka mixture and ion chromatography. Titanium dioxide (TiO_2) was used as a catalyst in the determination of nitrogen in coal by Banerjee (3B). He found that TiO_2 is a more efficient catalyst than selenium (Se) and gives results closer to the calculated values of nitrogen in coal. An ammonia-sensitive electrode was used by Rice et al. (15B) to determine nitrogen in coals. Their results agreed with those obtained by standard methods. The percentage of carbon in coals was investigated by Wind (21B) and Kalman (10B) using cross polarization (CP) magic angle spinning (MAS) nuclear magnetic resonance (NMR); the results of nuclear polarization experiments are presented, and the carbon percentage is detected by cross polarization.

Oxygen. Zischka and Stremming (22B) described an apparatus for the automated determination of oxygen (O) in coals and coal products. The results obtained are similar to those from standard methods (i.e., by difference). Mahajan (12B) used a fast neutron activation analyzer (FNAA) to determine the organic oxygen (O_{org}) in coal by subtracting the oxygen (O) in the mineral matter from the total oxygen (TO) in the coal ($\text{O}_{\text{org}} = \text{TO} - \text{O}$). Saito and Morohashi (16B) used gas chromatography to determine oxygen in coal.

Sulfur. Sakurai (17B) discussed an apparatus for sulfur determination in coal and coke. The apparatus consists of a high-temperature furnace, supplied with oxygen for combustion of the sample, and a tube packed with water-absorbing material; the absorption of water eliminates low values of sulfur dioxide (SO_2) due to the water- SO_2 interaction. Two methods for on-line determination of sulfur were investigated, developed, and tested by Armstrong and Page (1B) and Page and Piggins (14B). The methods are FNAA with iron fluorescence measurements by Armstrong and XRF with iron fluorescence measurements by Page. Many of the values obtained by Page are acceptable for on-line monitoring. A simple automated colorimetric analysis for the determination of pyritic sulfur in coal is described by Whitaker and Bryant

(20B). Gills (7B) discussed the methods and procedures used at the National Bureau of Standards (NBS) to certify sulfur content in coal standard reference materials (SRM's). Duran et al. (5B) investigated the presence of elemental sulfur in coal samples. They found that sometimes elemental sulfur was 5% of the total sulfur in a coal sample, but coals from the Premium Coal Sample Program were free of elemental sulfur. This observation indicated that elemental sulfur does not occur naturally in coal but is produced after coal is exposed to air.

Forms of Sulfur. Heunisch (8B) discussed the advantages and disadvantages of the various methods (e.g., ASTM D2492, X-ray diffraction (XRD), Mössbauer spectroscopy, and DTA) for the determination of pyritic sulfur in coal. A method for the direct determination of organic sulfur in coal has been developed by Hsieh and Wert (9B) using transmission electron microscopy (TEM) with an energy-dispersive X-ray detector. This technique is also used to detect spatial variation of sulfur over small distances within the coal maceral. Fully automated determination of organic sulfur in coal was performed by Timmer et al. (18B) using scanning electron microscopy (SEM) combined with X-ray microanalysis (XRMA). The values yielded by this method agree well with those obtained by using standard methods for low-pyrite coals. The results for high-pyrite coal using SEM/XRMA are higher than those from the standard methods values. The discrepancy may be caused by incomplete extraction of pyrite inclusions in the wet analysis of coals. Ban et al. (2B) compared the ASTM methods to the X-ray fluorescence determination of sulfur in coals. The results agreed for sulfide sulfur, combustible sulfur, organic sulfur, and pyritic sulfur. The sulfate sulfur values were lower for the ASTM method. Warne (19B) used thermomagnetometry-thermogravimetry (TM-TG) to determine the pyrite in coal.

INORGANIC CONSTITUENTS

Minerals. Three methods [i.e., DTA, TGA, and differential thermogravimetry (DTG)] were used to recognize patterns in the mineral mixtures in several coals from Eastern Kentucky and Southwestern Illinois. The concepts of both atmospheric interaction and mineral-mineral interaction and their effects on the final thermal analysis pattern are addressed by Earnest et al. (6C). Kiss et al. (12C) studied the distribution of minerals, inorganics, and sulfur in brown coal; the salts that dissolved in the aquifer water and the original salts from coalified-plant material contributed to the distribution of metals and sulfur in the Australian coal seams. Ruppert et al. (32C) found that 76% of the quartz in Upper Freeport coal was nonluminescent in the visible range and is therefore assumed to be authigenic (formed in place).

The discharge-excited low-temperature-ashing (LTA) technique was used to separate mineral matter from coals. A new technique developed by Adolphi et al. (1C) shortens the ashing time of coal (i.e., 4 h for lignites and subbituminous, and 8 h for bituminous and semianthracite). The isolated minerals can be analyzed by using a combination of XRD and electron microscopy. Kister et al. (13C) showed the importance of sample preparation and grinding in the analysis of minerals in coals. The coals were analyzed by IR spectroscopy, and LTA samples were analyzed by successive spectral subtraction of reference minerals that had been identified by XRD. Tsunashima and Yoshida (42C) reviewed 81 references on identification and quantification of mineral matter (e.g., pyrite and kaolinite) in coal ash by XRD and IR spectroscopy of LTA and by chemical transformations upon heating. Richardson et al. (29C) studied the microwave radiation of clay minerals in coal. Minerals such as kaolinite, illite, and montmorillonite were treated with 30–50% aqueous NaOH at room temperature, then were heated at 105 °C or subjected to microwaves. No reaction products were formed at room temperature. When the samples were heated, zeolite-like minerals formed from montmorillonite, and hydroxysodalite formed from kaolinite. With the microwave radiation, a hydroxysodalite-hydroxycancrinite mixture formed from kaolinite, nepheline formed from illite, and montmorillonite dehydrated.

Cox et al. (5C) estimated the inorganic-to-organic chlorine ratio in coal by using dimethyl sulfoxide (Me₂SO) with 0.1 M KNO₃, the ASTM D 2361 (Chlorine in Coal) method for

extractions, and a biamprometric titration for analysis.

Identification of the iron-bearing minerals [i.e., pyrite, FeSO₄, Fe₂(SO₄)₃, jarosite, and variously hydrated FeSO₄] was studied by Audley et al. (2C), Pankhurst et al. (25C), and Taneja and Jones (40C) using Mössbauer spectroscopy. The results agree with those using standard methods. The influence of iron on the NMR was studied by Lynch et al. (19C). They showed that magnetic ordering of such dispersed paramagnetic ions can result in a significant proportion of the proton populations being NMR invisible.

Hull and Horlick (9C) developed a electrothermal vaporization sample introduction system for inductively coupled plasma atomic emission spectroscopy (ICP-AES) that allows direct analysis of solids, powders, and small volumes of solutions and eliminates preparation of coal, coke, and coal-derived samples. Pearce et al. (26C) and Sato and Sakata (33C) determined many elements in coal ash by ICP-AES, although they did not use the Hull and Horlick device, and their sample preparations were not the same.

Timmer et al. (41C) combined energy dispersive X-ray analysis and SEM to quantitatively and simultaneously analyze eight elements in coal: Al, Si, S, Cl, K, Ca, Ti, and Fe. The method was applied to 16 types of coal and two samples of fluid-bed ash.

Trace Elements. A study of Canadian coals by Goodarzi et al. (8C) showed that local conditions had considerable influence on the concentration of certain trace elements. They studied both low- and high-rank coals using XRF and NAA. Lindahl et al. (18C) studied U.S. coals and concluded that cooperation between chemists and geologists was needed to eliminate anomalous data. Limic et al. (17C) and Raask (28C) reviewed the occurrence and concentration of trace elements in coal. The distribution of trace elements in U.S. coals was studied by Palmer et al. (24C).

The XRF and ICP-AES analyzers were used by Norton et al. (23C) to determine trace elements in coals and treated coals. Treatment of coal with aqueous Na₂CO₃ reduced the levels of Mn, Pb, and Zn by greater than 75%. Coals treated with molten caustic showed substantial reduction in the concentration of As, Ce, Hg, Pb, Rb, Se, Sr, and Zn.

Nakashima et al. (22C) and Rigin (30C), used atomic absorption spectroscopy (AAS) to analyze digested coal samples. Rigin digested his samples with the following four methods: Li₂BO₇, K₂O₂, acid digestion, or autoclave fluorination with XeF₄. Nakashima studied acid digestion. Rapid dissolution by microwave heating, followed by ICP-AES analysis, was employed by Nadkarni (21C). The method was tested on a variety of standard reference materials (SRMs) from the NBS and yielded reproducible and accurate results. Using AAS and ICP-AES and SRMs from NBS, Scholz et al. (35C) did a round-robin study to determine the accuracy and precision of trace element analyses done on coal, slag, ash, and pure gas samples. Simultaneous determination of Al, Fe, Ca, K, Mg, Ti, Na, Ba, Sr, P, Mn, Zn, Pb, Cr, Cu, Ni, La, Y, Co, and Sc was accomplished by Haraugchi et al. (8C) using acid digestion and ICP-AES analysis. Mogi et al. (20C) studied major and trace elements in coal ash by using a combination of XRF and ICP-AES. The relative error for major elements was less than 10% and for trace elements less than 20%. To study the concentration of 42 trace elements in Canadian coal samples, Landheer et al. (15C) employed NAA, AAS, and ICP-AES analyses. African coals were analyzed for boron using ICP-AES by Pougnet and Orren (27C). Willis and Hart (43C) combined both NAA and XRF to determine trace elements in coals, and the experimental errors were given for the determination of B, Be, F, Hg, Cd, and Tl. Nigerian coals were analyzed by Borishade et al. (4C) using three methods (i.e., the ASTM, NAA, and FNAA). The coals were suitable for a blend with coking coals. The analyses proved accurate for most of the trace elements. Other researchers utilizing NAA for measuring trace elements of coal and coal fly ash were Jin et al. (11C), Roseberry and Dyer (31C), and Suzuki et al. (39C). Li (16C) determined trace elements measuring the short-lived radionuclides produced by FNAA. Bellido (3C) and Savanonda et al. (34C) used NAA to determine uranium in coal. Radioactivity in coal is of interest because of the environmental problems that may result from coal processes and by-products.

Sequential elution solvent chromatography (SESC) on an SiO₂ column was used to obtain 10 fractions of preasphaltenes

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(PA) from SRC-I. The fractions were then analyzed for Ti by Iskander et al. (10C) using a NAA. The use of γ activation analysis on brown coal and brown coal ashes is described by Stuchlik and Langrock (38C). The elements determined were No, Tl, Mo, and Pb, and their sensitivities are given. The determination of Ga in sediment, coal, and coal fly ash using AAS with a nickel-matrix modification was reported by Shan et al. (36C).

CALORIFIC ANALYSIS

A method for the simultaneous determination of calorific value, ash yield, and total moisture content of soft brown coals was developed by Kreft et al. (8D) using neutron thermalization and γ backscattering. They analyzed over 80 coal samples having calorific values up to 15.5 MJ/kg, ash yields of 3.6–76.1 wt %, and total moisture contents of 11.4–61.7 wt %. Dean and Chiu (5D) used ion chromatography combined with the ASTM D 3177 (bomb combustion) method to determine the calorific values of the same coals they analyzed for S, Cl, and F. A rapid, accurate, and nondestructive method for determining the calorific properties of coal samples was

developed by Amer (1D) using photothermal analysis.

Chien et al. (3D) and Brown et al. (2D) studied coal oxidation and found that the heating value of coal drops linearly as the carbonyl concentration increases. Fujii et al. (6D) compared vitrinite reflectance and sporinite fluorescence in relation to coal properties; the sporinite fluorescence is closely associated with calorific values, but vitrinite reflectance is more associated with volatile matter.

Calculation of the calorific content of coals using ultimate and proximate analyses was discussed by Given et al. (7D) and Chyi and Quick (4D), respectively. Both provide an equation related to the respective analysis. Chyi also gives an equation that holds for oxidized coals and a corollary to describe the effect of coal preparation on the calorific value.

PETROGRAPHY

Chandra (7E) discussed the origin and postdepositional events in coals as important parameters for their classification. The classification of Chinese coal for power generation was discussed by Sun and Liu (16E), while Detaevner et al. (8E) reported on the classification of coals using their elemental and mineral composition to predict furnace fouling and slagging. Tumuluri and Shrikhande (17E) developed codification charts for classifying Indian coals from proximate analysis from moisture or cokability data. Snyman et al. (15E) proposed a classification system for South African coals from chemical and petrographic characteristics. Ogata et al. (11E) evaluated the use of electron spin resonance (ESR) parameters for the classification of coal. The relationship between vitrinite reflectance and coal rank was discussed by Uribe and Perez (18E) and by Radke et al. (14E). Posil'nyi and Kurakov (13E) distinguished four groups of anthracite with TGA and dilatometry, while Meuzelaar et al. (10E) used pyrolysis and mass spectrometry to reveal structural differences between lignites.

Laser microanalysis was used by Barea and Panaitescu (4E) to determine the petrographic composition and the distribution of Si, Fe, Al, Ca, Mg, Ti, and nine minor elements in three Romanian coals. Dyrkacz et al. (9E) used density gradient procedures to provide information on the differences both between macerals and across each maceral group density band. Bertrand (6E) concluded that oil potential might better be evaluated from the proportions of various vitrinite macerals than by the proportion of exinite in coal.

Maceral composition and coal rank determination by reflectance analysis were reported by Parkash et al. (12E) using an automated, computerized Zonax system coupled with a Zeiss reflectance microscope. A microscopic step-scanning technique that eliminates the irregular effect of the reflectivity around the border of the coal was used by the Nippon Steel Chemical Co., Ltd. (2E), to measure the amount and average reflectivity of vitrinite in briquets formed with coal powder and binder. Stepwise scanning of a coal specimen with a polarized microscope was reported by the Sumitomo Metal Industries, Ltd. (3E), for automatic analysis of microstructure of coal for metallurgical coke manufacture. Hoofdgroep Maatschappelijke Technol. (1E) used SEM and X-ray microanalysis to show that two coals had minerals intimately mixed with the macerals, while a third coal had minerals, including FeS, in a markedly layered structure. Beny-Bassez and Rouzand (5E) used optical microscopy, transmission electron microscopy, and Raman microspectroscopy to study the elimination of "defects", such as heteroatoms and tetrahedral carbons, from a graphitizable series as coals are heated or as coals increase in rank.

PHYSICAL METHODS

Parkash et al. (6F) studied the porosity of representative Alberta subbituminous coals with gas adsorption, He and Hg displacement, and Hg porosimetry. Helium densities (1.372–1.468 g/cm³) were lower than the methanol densities (1.522–1.696 g/cm³), and both were higher than *n*-hexane densities (1.323–1.371 g/cm³). A new method for measuring the equilibrium swelling ratio and initial swelling rate of coal in a variety of solvents was reported by Aida and Squires (1F). Taits et al. (9F) determined the physicochemical properties, microporosity, and modulus of elasticity of coal by continuous indentation.

A new plastometer for measuring the apparent viscosity of plastic coals under rapid-heating, high-temperature conditions was developed by Fong et al. (3F). The initial coal-softening temperature was found to be insensitive to the heating rate. Read et al. (7F) used a pressurized Gieseler plastometer to show that the maximum fluidity of coal is affected by pressure and that increased pressure decreases the softening temperature and maximum fluid temperature and increases the resolidification temperature and total temperature range of fluidity. A group of 40 coals was examined by Lloyd et al. (4F) with regard to ASTM Gieseler plastometric properties, and 29 of the coals were also studied over a range of temperatures by isothermal Gieseler plastometry. The observed maximum fluidity was less consistent than maximum fluidities by slope intersection. Additional analytical data were used for a three-term expression, multiple linear regression analysis for estimating maximum fluidities.

A laboratory apparatus was designed by Mel'nichuk (5F) for differential thermal analysis (DTA) and the simultaneous measurement of electrical potential vs. time, flash point, and analysis of decomposition products during carbonization or pyrolysis of solid fuels (e.g., coal, semicoke, and coke). Fourteen low-rank coals were subjected to thermogravimetric-mass spectrometric (TG-MS) and pyrolysis-gas chromatography-mass spectrometric investigation by Blazso et al. (2F). The relative amounts of isoprenoid and aromatic marker compounds in the pyrograms at 600 °C were related to the rank of the coal. Rushev and Atanasov (8F) measured the activation energy of thermal decomposition of solid fuels (coals and lignites) from TGA and DTG analysis.

SPECTROSCOPY

Brown et al. (3G) found good correlations in the fluorescence of inertinite and vitrinite with coal rank, Gieseler fluidity, dilatation, g value, and crucible-swelling number. The wavelength of the maximum fluorescence intensity increases with rank and the intensity peaks at the transition from high- to medium-volatile coal. In the study of inertinite by fluorometry, Diessel (7G) preferred a blue exciter filter (450–490 nm) to the more intense signal received through a green exciter filter (546 nm) because the fluorescence spectrum is broader, the microscopic image more polychromatic, and the maceral identification easier. The method of quantitative fluorescence microscopy has only recently been applied to the study of coal macerals in situ. To avoid the nomenclature problems caused by the abundance of new maceral varieties identified with this technique, Crelling (5G) developed the concept of spectral type. The fluorescence properties of fluorinite, sporinite, and cutinite showed systematic change with an increase in coal rank.

The FT-IR spectrum of coal was evaluated by Fredericks et al. (12G) using factor analysis to condense the data followed by the use of multiple linear regression for correlating with a calibration set of well-characterized samples. The FT-IR difference spectroscopy was used by Gethner (16G) to evaluate the effects of 100 °C vacuum drying on Rawhide subbituminous coal. The substantial IR absorptivity changes implied that significant perturbation of the organic structure takes place upon drying. Starsinic et al. (29G) used FT-IR to study acid-demineralized and cation-loaded lignites to determine that exchange methods now in use to determine that carboxyl groups in coal produce systematic errors. Diffuse reflectance infrared spectroscopy (DRIS) was described by Fuller and Smyrl (13G) as a simple, fast, versatile, and informative method for coal analysis. Fysh et al. (14G) noted that quantitative near-IR diffuse reflectance spectroscopy is restricted by the transparency of quartz but is sensitive to the aliphatic structure of coal. The technique of Fourier self-deconvolution was applied by Wang and Griffiths (32G) to reduce the widths of all bands in the DRIS spectra of coals. They were able to resolve several bands between 3000 and 2800 cm^{-1} that are due to C-H stretching modes of alkyl groups in different environments.

Silbernagel et al. (28G) studied exinite, vitrinite, and inertinite maceral samples isolated from coals of varying rank with ESR. Carbon radical g values decreased with increasing coal rank for both vitrinite and exinite in association with a loss of oxygen at higher rank. There was also an increase in line widths, radical densities, and ability to absorb microwave

radiation with coal rank. Electron spin-echo spectrometry (ESE) was employed by Doetschman and Mustafi (8G) to isolate the vitrain radical spectrum in bituminous coals from relaxation time differences between macerals. The echo decay was modulated at frequencies of 2.5 and 5 MHz, and a model using the predicted echo modulation was also able to predict most of the electron-nuclear double resonance (ENDOR) lines. Evidence that the coal radical spin multiplicity is doublet, $S = 1/2$, was presented, and echo modulations were consistent with hyperfine interactions in a perinaphthyl or similar type of radical oriented in coal layers. The quenching of paramagnetic centers in coal during extraction with THF, pyridine, or ethylenediamine was attributed by Duber and Wieckowski (10G) to the destruction of intermolecular donor-acceptor bonds between the two molecular phases of coal. Malhotra and Graham (22G) detected magnetite particles in coal with ferromagnetic resonance (FMR). The potential usefulness of FMR in the identification and physical and chemical characterization of ferro- and ferrimagnetic minerals in coal was discussed.

A review of solid-state, cross polarization, magic-angle spinning, nuclear magnetic resonance (CP-MAS-NMR) carbon 13 methods for coal was given by Tekely and Delpuech (31G), while discussions on this topic were presented by Gerhards and Kasueschke (15G) and by Maciel (21G). The past few years have seen considerable success in revealing structural detail from CP-MAS-NMR spectra of low-rank coals, especially from the aliphatic peak region; Jancke et al. (19G) found that aliphatic carbon in brown coals exists as long-chain paraffinic side arms, while Newman and Davenport (25G) used a combination of high magnetic field (4.7 T) and resolution enhancement to extract additional spectral details with 32 low-rank coals from New Zealand and Australia. Signal heights were used to characterize organic functional distributions. A decrease in CP-MAS-NMR aromaticities with increasing coal rank was found by Sfihi et al. (27G), by Botto et al. (2G), and by Davenport and Newman (6G), although the latter authors did not feel that aromaticity was a useful measure of rank, preferring the correlation to phenolic carbon. Zilm and Webb (33G) applied carbon-13-proton chemical shift correlation spectroscopy in assigning carbon-13 signals from the CP-MAS-NMR spectrum of a coal. The CP-MAS-NMR relaxation time behavior for Alberta subbituminous macerals was extensively characterized by Axelson and Parkash (1G). The proportion of bridgehead aromatic carbons in coals was determined by Burgar et al. (4G) using CP-MAS-NMR, while Pugmire et al. (26G) used the dipolar diphasing technique in the evaluation of protonized and nonprotonized carbon in coal and vitrinite. A novel NMR method for determining the aromaticity of coal was evaluated by Jones et al. (20G) and involves polarization transfer from the paramagnetic electrons to enhance the intensity of the carbon-13 NMR spectrum. The potential value of double-cross-polarization $\text{H}-^{31}\text{P}-^{13}\text{C}$ NMR to structure and reactivity information in coal and *o*-methoxydiphenylphosphine-derivatized coal was reported by Hagaman et al. (18G). Tekely et al. (30G) eliminated unwanted overlapping of aromatic side bands with the aliphatic region of CP-MAS-NMR spectrum of coal by selective saturation of the aromatic signals of coal.

Secondary ion mass spectrometry (SIMS) and X-ray spectrometries have generally been applied to the inorganic constituents in coal but have been recently applied to whole coal and coal macerals as well. The structures of demineralized vitrinites from anthracites, coking coals, and gas (high-volatile bituminous A) coals were examined by Grigoriew and Diduszko (17G) by XRD. Fourier-transform graphs were presented and coordination numbers were calculated. Dou et al. (9G) discussed the use of computed tomography in studying the internal structure of coal. Small angle X-ray scattering (SAXS) was applied by Foster and Jensen (11G) to measure the specific surface area of coal and to infer a pore size distribution. Martin et al. (23G) used SIMS to reveal elemental associations in mineral matter of coal and in the organic-inorganic phases. The surface adsorption of oxygen-18 was also studied and found to be reversible with fusinite, which, despite its lower reactivity, absorbed more oxygen than vitrinite. Martin et al. (24G) distinguished organic and inorganic domains of coal macerals with SIMS imaging.

COKING PROCESS AND COKE TESTING

Methods for evaluating the quality of coke manufactured at high temperatures were evaluated by Forizs et al. (6H). Correlations were discussed for methods to determine strength and particle size distribution. Lin and Chin (15H) found that a particle size cut of 40–60 mm gave the best representative data for total coke analysis, including mechanical strength, reactivity, porosity, and structure. Coke quality and its assessment were discussed by Goleczka and Tucker (9H).

Golec and Bysiewicz (8H) developed an automatic apparatus to determine coke reactivity in accordance with the BN 78/0511-31 standard. The concentration of CO₂ in offgases from the oxidation of coke under standard conditions was used as a measure of coke reactivity. Gladkov et al. (7H) measured coke reactivity by rotating a sample in a drum of 1000 °C or less and then treating the sample with CO₂ for determination of changes in size fraction and chemical activity. A regression equation was obtained by Shi and Wang (21H) for describing coke reactivity, porosity, and microstructure. Coke reactivity was determined from a quadratic fitting equation and isolines graph. Hung and Chiu (11H) observed that the reactivity of coal in coking can be used as an index to the extent of previous oxidation but does not affect the cold strength of coke. The maximum weight loss rate in the TGA of oxidized coals was related by Izuhara et al. (12H) to the CO₂ reactivity of coke prepared from such coals. Rod'kin et al. (20H) described the conditions under which the DTA and DTG analysis of coke in an oxygen-containing atmosphere could be measured without interference from the diffusion rate of oxygen.

Coke and semicoke structures were evaluated, in terms of the distribution of an anisotropy index, by Nippon Kokan K.K. (1H) from the microscopically measured maximum and minimum reflectivities using a rotating polarizing plate. Brown et al. (5H) found microtextural analysis invaluable in evaluating cokes for blast furnace use. The Sumitomo Metal Industries, Ltd. (2H), described the use of a polarizing microscope for the automatic analysis of the microstructure of coal used in the manufacture of coke. The microscopic examination of cokes, obtained by coal coking at 1000 °C with coal extracts, was used by Jasienko and Gryglewicz (13H) to classify coals. Qian et al. (18H) evaluated the bonding of solid additives in cokes with optical microscopy and SEM. Changes in the coal/coke structure of polished coals carbonized in air or CO₂ were studied by Hachiya and Mastsumura (10H) using a specially constructed hot stage for a polarizing microscope.

Roberts (19H) concluded that a simple second-degree regression equation in coal rank and type variables is better for predicting coke strength for Australian coals than petrographic data alone, while Bergmann (4H) used mineralogy and petrographic data of coals to evaluate coking properties. A discussion by Stuchlik (22H) of various methods of coke analysis advocated petrographic and plastometric methods for evaluating coke for metallurgical uses. An improved method for predicting the strength of coke from inertinite-rich coals was proposed by Pearson and Price (17H) and is based upon an equation involving the maximum coal reflectance value. Li (14H) concluded that coal rank, as determined primarily from reflectance, is the main factor influencing the strength of coke used for China Steel.

When ash was determined in coke with the X-ray backscattering technique by Pandey and Prasad (16H), the variations in measurement were within allowable limits as long as moisture levels were below 5% by weight. Belonogova et al. (3H) used X-ray diffractography of ash, semicoke, and coke to follow the fate of clays in coking.

MISCELLANEOUS

The relation between number-average molecular weight (M_n) and weight-average molecular weight (M_w) was examined by Collins et al. (5I), and it was demonstrated how this can be employed to determine the M_w of chemically treated coals. Their newly calculated M_w (3300) did not agree with Sun and Burk's value of 7882, and they concluded that Sun and Burk's value was wrong because their method of calculation was incorrect. Their comparison of method and calculation on Katz's data showed agreement. Parkash (8I) studied true density of coal using the Micromeritics helium autopycnom-

eter. The coal samples (60 mesh) were previously dried in a vacuum oven at 100 °C. The sample composition was determined by using the following manufacturers instruments: Fisher for moisture, ash, and volatile matter; LECO for sulfur; and Perkin-Elmer for C, H, N, and O. The true densities appear to increase linearly as the percent of dry ash concentration increases. Parkash concluded that the true densities of low-rank coals, maceral concentrates, and chars are related to their chemical composition.

Chen and Pagano (4I) studied chlorine removal from Illinois coal and found that it slightly improved the quality of coal. Soong et al. (9I) used an ion-selective electrode method to determine fluorine (F) in lignites and in subbituminous and bituminous coals. They found that New Zealand coals are lower in fluorine than other coals.

Homogeneity in coal was investigated by Bhagat (1I) using the fracture toughness analysis and tensile strength analysis of eastern coals from the Pittsburgh area. The conclusion was that a combination of high fracture toughness data and high tensile strength data indicated homogeneity in coal.

Many trace and minor elements are present in some characterized coal macerals, and McIntyre et al. (6I) used SIMS to measure qualitatively the distribution of these elements in vitrinite, fusinite, and exinite coal macerals. X-ray photoelectron spectroscopy (XPS) was used to identify the elevated concentrations of organofluorine compound in fusinite, and a widely dispersed inorganic aluminosilicate phase was identified in vitrinite samples using SIMS. They found that exinite contained lower concentrations of most elements compared to the other macerals.

The origin of polymethylene moieties was determined by Calkins and Spackman (3I) using NMR spectroscopy. They found that the original plants in the formation of the coal are the source for polymethylene components in coal.

Bisselle and Brown (2I) reported on the collected computerized data on the concentrations of radionuclides in U.S. coals. The emphasis was on U levels in coals used in power plants to determine if there was an environmental problem. They found that a better identification and selection of coals for power plants could eliminate the environmental problem.

The thermochemistry of coal oxidation was studied on five types of Polish bituminous coals by Matyjaszczyk and Przeliorz (7I) using TGA, DTG, DTA, and evolving gas analysis (EGA) simultaneously. They investigated nonisothermal coal oxidation. The results characterized the tendency of a given coal toward oxidation. Oxidation is a problem in coking coal.

STANDARD METHODS

The development and standardization of test procedures for coal and coke are coordinated by ASTM (1J) through the D5 committee. Standards that were adopted, revised, or reapproved during the period covered by this review include the following: D121-85, Standard Definitions of Terms Relating to Coal and Coke; D409-85, Grindability of Coal by the Hargrove-Machine Method; D2014-85, Expansion or Contraction of Coal by the Sole-Heated Oven; D1412-85, Equilibrium Moisture of Coal at 96% to 97% Relative Humidity and 30 °C; D3173-85, Moisture in the Analysis Sample of Coal and Coke; D2797-85, Preparing Coal Samples for Microscopical Analysis by Reflected Light; D2798-85, Microscopical Determination of the Reflectance of the Organic Components in a Polished Specimen of Coal; D2799-86, Microscopical Determination of Volume Percent of Physical Components of Coal; D2015-85, Test Method for Gross Calorific Value of Coal and Coke by the Adiabatic Bomb Calorimeter; D3286-85, Gross Calorific Value of Coal and Coke by the Isothermal Jacket Bomb Calorimeter; D2361-85, Test Method for Chlorine in Coal; D4239-85, Sulfur in the Analysis Sample of Coal and Coke using High Temperature Tube Furnace Combustion Method; D4326-84, Major and Minor Elements in Coal and Coke Ash by X-ray Fluorescence; D3761-84, Total Fluorine in Coal by Oxygen Bomb Combustion/Ion Selective Electrode Method; D388-84, Coals by Rank; and D2639-85, Plastic Properties of Coal by the Constant-Torque Gieseler Plastometer.

Standards that were covered by this review with no revision are the following: D4181-82, Evaluation of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and

Coke; D2013-78, Preparing Coal Samples for Analysis; D720-83, Free-Swelling Index of Coal; D2492-84, Forms of Sulfur in Coal; D3302-82, Total Moisture in Coal; D3682-83, Major and Minor Elements in Coal and Coke Ash by AAS; D3683-83, Trace Elements of Coal and Coke Ash by AAS; D2795-84, Analysis of Coal and Coke Ash; D1756-84, Carbon Dioxide in Coal; D3177-84, Total Sulfur in the Analysis Sample of Coal and Coke; D3179-84, Nitrogen in the Analysis Sample of Coal and Coke; D3180-84, Calculating Coal and Coke Analyses from As-Determined to Different Bases; D3178-84, Carbon and Hydrogen in the Analysis Sample of Coal and Coke; D3176-84, Ultimate Analysis of Coal and Coke; D4208-83, Total Chlorine in Coal by the Oxygen Bomb/Ion Selective Electrode Method; D2796-82, Definition of Terms Relating to Megascopic Description of Coal and Coke Seams and Microscopic Description and Analysis of Coal; D3175-82, Volatile Matter in the Analysis Sample of Coal and Coke; and D197-82, Sampling and Fitness 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 Trusell (6K). A review of gas chromatographic methods suitable for the analysis of natural gas was given by Willis (7K).

Techniques for the measurement of liquefied petroleum gas were reviewed by Caffey (1K). Cox (2K) reviewed the fundamentals of gas chromatography in relationship to the analysis of natural gas. Tramel (5K) reviewed hygrometers and other techniques for measuring the moisture content of natural gas. Jamieson and Sikkenga (3K) evaluated commercial equipment for on-line measurement of water dew point in natural gas. They concluded that a better understanding of the behavior of water vapor in high-pressure natural gas is needed. The relationship of thermodynamics and equations of state to the measurement of natural-gas flow was reviewed by Starling (4K).

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 Clement et al. (3L) in 1986. This review covers only those publications directly related to gaseous fuels.

Quantitative gas chromatographic techniques for natural gas and natural-gas liquids were discussed by McCann and Cockran (7L). Standardization and the reasons for such determinations were included in the discussion. Ruchnov and Fatkudinova (8L) described systems for the chromatographic analysis of natural-gas mixtures. Natural gas was analyzed by Vlckova and Kavan (10L) using three gas chromatographic columns packed with DC 2000, Poropak Q, and 13X molecular sieve and operated isothermally at 70 °C. In a second method, the higher hydrocarbons were determined using a 40 to 135 °C heating program on a column packed with UCW982. The two methods together found 34 C₁₋₉ hydrocarbons, of which 27 were identified. Mariich et al. (6L) determined the composition of coke oven gas with a rapid gas chromatographic procedure for O, H, N, CO, CO₂, CH₄, C₂H₄, and C₂H₆. Excellent separation was achieved with 3-m by 3-mm columns filled with either Zeolite CaA or Polysorb 1 at 40 °C and using a katharometer detector and He or Ar carrier gas flowing at 60 mL/min. A fused-silica column (15 m long by 0.42 mm i.d.) packed with Chromosorb 102 (90–100 μm) was used by Al-Thamir (1L) to analyze natural gas for CO₂, H₂S, and C₁₋₆ hydrocarbons. Helium carrier gas was used with 20 °C per minute temperature programming from 50 to 110 °C after an initial 5-min hold.

Saadat and McKiney (9L) described a portable micro gas chromatograph (Micromonitor) for the determination in 2 min of natural-gas composition (including C₆₋₈ fractions), Btu content, specific gravity, and compressibility. The system is suitable for field applications and does not use back flush valves. A portable, automatic gas chromatograph for the analysis of natural gas was described by Hawker and McKiney (4L). It measures only 2 by 4 by 4.5 in. and is fabricated by the same micromachining technology that is used for integrated circuits.

A new polysiloxane gum was synthesized by Kuei et al. (5L) and was employed as the stationary phase in the analysis of natural gas by capillary-column gas chromatography. The new phase is a 50% *n*-octylmethylpolysiloxane and demonstrates high efficiency and excellent cross-linking properties that improve the resolution for volatile hydrocarbons when compared to OV-1. Chen et al. (2L) analyzed natural gas using a porous-layer, open-tube capillary gas chromatographic column (0.28 mm by 12 m) in which the inside was coated with five layers of Zeolite 13X with a thickness of 0.4 mg/cm² (9.4 μm). The equivalent of 2100 theoretical plates was observed in the analysis of C₃H₈. The same column was used to determine O, CO₂, and water.

SULFUR COMPOUNDS

Dolzanskaya (5M) reviewed contemporary methods for determining hydrogen sulfide in coke-oven gas. The methods involved precipitation of H₂S followed by turbidimetry, photocolormetry, gas chromatography, and various electrochemical procedures. The hydrogen sulfide content of natural gas was determined by Mikhal'kov et al. (10M) by introducing 10 mL of gas containing more than 1% H₂S into a column packed with (OAc)₂Pb-impregnated silica gel and measuring the height of the discolored silica gel layer. The device may be modified for use at low H₂S concentrations, in natural-gas wells, or for the simultaneous determination of mercaptan and H₂S. Akhmadullina et al. (1M) examined the GOST 22986 procedure for determining total sulfur in refinery gases and pentane fractions by combustion, absorption, and titration. They found the presence of NO_x caused serious errors that could be reduced by substituting a turbidimetry measurement for titration. Inorganic sulfur in water gas was determined indirectly by Chen and Fan (3M). Sulfur compounds were reduced to S²⁻ by using an Auger reagent of 3 g of KI and 2 g of H₃PO₃ added to 20 g of H₃PO₄. The S²⁻ was precipitated with excess Hg²⁺, the excess Hg²⁺ was reduced to Hg with SnCl₂, and the Hg was determined by atomic fluorescence spectrometry. Results were comparable to the methylene blue method.

Dementii et al. (4M) used mathematical analysis to optimize the design of an ionometric analyzer for determining the mercaptan content of natural gas. An aqueous Br₂ solution was sprayed into the gas stream, and the outgoing liquid was analyzed with a bromide-selective electrode. A natural-gas analyzer, based upon coulometric titrimetry, for determining concentrations of thiols, organic sulfides, organic disulfides, and H₂S was reported by Vincent et al. (13M). Separation was with three scrubbers containing 1% CdSO₄-2% H₃BO₃, 10% NaOH, and 0.5% AgNO₃, respectively, and a fourth that was empty. Akhmadullina et al. (2M) determined H₂S, mercaptans, and COS in a petroleum refining gas by consecutively bubbling the gas through 3 wt % aqueous Na₂CO₃, 40% aqueous NaOH, and 5% alcoholic MeNH₂Et, respectively. The aqueous solutions were then titrated potentiometrically with a AgNO₃-NH₃ complex for H₂S and mercaptans, and COS was determined photometrically. Jedrzejczyk and Pasykiewicz (8M) described two new methods for the determination of sulfur compounds in fuel gases. One method involves the chemisorption of H₂S and mercaptans in concentrated aqueous KOH and of COS in alcoholic H₂NCH₂CH₂OH, followed by automatic potentiometric titration using AgNO₃ solution. In the other method, H₂S and C₁₋₄ mercaptans were chromatographically separated on a column packed with Chromosorb W containing 20% nonyl phthalate. Measurement was in an electrochemical cell containing 10% CrO₄²⁻ [for reduction of Cr(VI) to Cr(III)].

Gibbons (6M) monitored EtSH, *t*-BuSH, MeSEt, and Et₂S in British natural gas by using chromatographic separation and a flame photometric detector (FPD). Macak et al. (9M)

determined H_2S , RSH ($R = \text{alkyl}$), RSR , and $RSSR$ in natural gases of the USSR by using gas chromatographic separation and FPD. An O to H ratio of 0.250 gave the maximum detector response, and the optimum detector temperature was 125 °C. Polyphenyl ether was used for separating low-molecular-weight components, and Triton X305 was used for separating sulfides and disulfides. Huber and Obbens (7M) used a novel combination of packed and capillary columns and a fully automated gas chromatographic system with FPD for the on-line monitoring (every 6 min) of H_2S , COS, and tetrahydrothiophene in natural gas. An apparatus for the determination of tetrahydrothiophene in town gas was described by Nardini (11M) and by Odajima et al. (12M).

CONDENSATES

In a review of chromatographic methods for the analysis of natural gas condensates, McCann (8N) included industry standards, sample handling, instrumentation, and calculations. The analysis of components in unfractionated natural-gas condensates was performed by Lunskii (6N, 7N) using two capillary columns kept at different temperatures (50 and 70 °C). Triska et al. (11N) used capillary gas chromatography and mass spectrometry to analyze the composition of a high-boiling natural-gas condensate from a deep well. Lisitsyn and Gorshkov (5N) analyzed alkylbenzenes and alkyl-naphthalenes in natural-gas condensates using capillary gas chromatography with two columns and an ultraviolet-light photometric detector. Thiophenes in the raffinate from the sulfuric acid extraction of a natural-gas condensate were determined by Agadzhanova et al. (2N) using column chromatography and mass spectroscopy. Pavlova (9N) determined water in gas-condensate hydrocarbons, C_{1-6} , with a gas chromatograph.

Boyle et al. (4N) described an apparatus for the continuous detection and monitoring of the hydrocarbon dew point of natural gas. The dew point of fuel gases was measured by Belski et al. (3N) using an electronic condensation hygrometer. The application of the Peng-Robinson equation of state to the calculation of natural gas condensate yields was tested by Tkachenko et al. (10N) and gave good agreement to experimental values at moderate pressures. Abbasov (1N) presented a method for calculating the pseudocritical temperatures and pressures of the C_{5+} components of natural gas condensates from their molecular weights and oil content.

CALORIMETRY

The Yamatake-Honeywell Co., Ltd. (2P), described a simplified calorimeter where a diluted fuel gas is heated to combustion, and the upstream and downstream temperatures are read. Temperature sensors at the points of entry of the fuel gas and oxidant and at the outlet for the combustion gases were used by Solov'ev et al. (13P) for the continuous measurement of the heat of combustion of a fuel gas. Todt et al. (14P) considered the heat from the combustion of a gas in a flowing heat-transfer solid to be proportional to the Wobbe index. A calorimeter was presented by the Yamatake-Honeywell Co., Ltd. (3P), where as preheated town gas is catalytically combusted and the temperature measured. Heitz (10P) measured the calorific value of a fuel gas by measuring the oxygen content, with a zirconium oxide (ZrO_2) sensor, before and after combustion with an oxidant gas. The zirconium oxide sensor was determined by Trimbos et al. (15P) to be the most successful technique to measure the oxygen content of exhaust gas from stoichiometric-type combustion calorimeters. Bohl et al. (5P) measured the oxygen content following the combustion of a compressed fuel gas with compressed air and related this to the Wobbe index. An apparatus with a double piston for measuring and feeding a fuel gas and excess air to a combustion chamber was described by Bergman (4P), where the calorific value of the gas was determined by measuring the oxygen content of the combustion gases with an electrode.

The use of on-line gas chromatographs in determining the calorific value of natural gas was reviewed by Foundos and Kersey (7P). Haas (8P) discussed the potential of combining gas chromatography and the flow computer, used in accurate orifice calculations, into a "total energy measurement system". An on-line micro gas chromatograph based upon an analyzer

micromachined from silicon wafers was described by Mowry (12P) as having an analysis time of 2 min. The system contains three coupled gas chromatographs, a microthermal conductivity detector, and a computer. van Rossum and Koning (16P) considered the question of how many components of natural gas are necessary in the calculation of calorific value.

The heating value of fuel gases was calculated from mass, volume, and ideal-gas-flow rates by Hall et al. (9P) and from flue gas composition by Mikhailov (11P). Flowers (6P) gave a calculator program that estimates natural gas calorific value, specific gravity, and condensable liquid content from the gas composition of mixed streams. The amount of volatile matter and the heating value of feed coal for coking were used by the Kawasaki Steep Corp. (1P) to estimate the heating value of the fuel gas, coke, and tar produced.

METERING AND DENSITY

Hoglund (5Q) reexamined the hydraulic and thermodynamic concepts of natural-gas flow through orifice meters in attempting to revise Report No. 3 of the American Gas Association. A new method for calibrating mass flowmeters was described by Singh and Puster (7Q) and is based upon matching the partial pressure of oxygen in combustion products of natural gas burned in oxygen-enriched air with that in normal air. Results were in good agreement with independent procedures and for natural-gas test samples. The technique was successful in measurements of differences as low as 1% of the effective hydrocarbon content. Ellington et al. (2Q) used an automated Burnett-type Z-meter to measure variations in the natural-gas compressibility factor to correct expensive errors in gas metering.

Densities of a calibrating gas (methane) determined by a swing process in absorbers (used with the metering of natural gas) were shown by Hinze and Jaeschke (4Q) to vary by 0.5% from direct densities determined by weight differences in a Ti-Al-V balloon flask. The velocities of sound in the calibrating and natural gas were used to bring agreement to within 0.1%. The equation used for making this correction was described in a separate article by Jaeschke (6Q). Tanaka (8Q) used a densitometer with an elastic material that has a resonant frequency that varies with the density of the surrounding fluid to detect phase separations and to measure the storage level of liquefied natural gas. Three models of equations of state were evaluated by Galicia and Leiva (3Q) for predicting the densities of liquefied-natural-gas mixtures. The COSTALD correlation was shown by Thomson and Hankinson (9Q) to be quite accurate and relatively simple to use for custody transfer of natural-gas liquid. Ellington (1Q) reviewed equations of state in relation to the measurement of natural-gas flow.

SAMPLING

An improved procedure was given by Johnson (2R) for sampling natural gas for pipeline liquids problems. Natural-gas condensable content was calculated following analysis by gas chromatography. Eight methods of natural-gas sampling were evaluated by Hefley et al. (1R) using 24 h independent laboratories. All of the methods tested gave good results, with the water displacement method giving the highest accuracy.

The sampling and analysis of natural gases from petroleum- and gas-containing basins were discussed by Vanyushin et al. (4R). Weres et al. (5R) described engineering tests performed on a sampler for geopressured gas wells. Auxiliary tools for effectively using the sampler were also described. The optimum conditions for taking representative natural-gas-condensate samples in a wellhead were determined by Ostrovskaya et al. (3R) at the boundary conditions of natural-gas flow (≥ 4 m/s) and differential pressure (≤ 10 –15%).

MISCELLANEOUS

Howind (10S) presented computer-assisted gasification monitoring and control technology, including various on-line GC-MS analyzers. The advantages of a boardless instrumentation system for coal gasification were described by Moriyama (16S), where flow diagrams, operating temperatures and pressures, etc., are displayed on cathode ray tubes. He

et al. (8S) used a two-column gas chromatograph to analyze coal gases. A method for measuring the gas outlet temperature in fixed-bed pressure gasification was given by Scjolz et al. (18S). Dilorio (6S) evaluated various radiometric techniques for measuring temperatures in coal gasifiers and concluded that optical-based methods will be unable to yield temperature information for the interior regions of gasifiers. Laser-based optical diagnostic techniques have been used in coal gasification by Oldenborg and Baughcum (17S) to monitor trace levels of alkali compounds and by Taylor et al. (25S) to measure species concentrations and temperature.

Advances in supercompressibility factor correlations for the natural-gas industry were described by Starling et al. (23S) and by Starling and Kumar (20S). The development of improved capabilities for computing supercompressibility factors used in the custody exchange of sweet, dehydrated natural gases was reported by Starling et al. (21S, 22S).

A highly sensitive natural-gas sensor described by Takenaka (24S) is based upon a sintered semiconductive $\alpha\text{-Fe}_2\text{O}_3$, whose electrical resistivity changes due to microscopic oxidation and reduction processes. Gas detectors based upon semiconductive SnO_2 elements were reported by Hitachi, Ltd. (2S), and by the New Cosmos Electric Co., Ltd. (3S), to show good selectivity for methane. Altpeter et al. (1S) investigated the feasibility of low-cost odorant test instruments based upon either a quartz piezoelectric crystal coated with a sorbent of AgNO_3 -Carbowax 1500 or a semiconductor device. The piezoelectric detector was irreversible and could only be used once. An optical device for determining ethane leaked from natural gas, LNG, or LPG pipelines that uses a wavelength that minimizes water and CO_2 interference was described by Shinohara et al. (19S). McRae (14S) discussed the possibility of backscatter absorption gas imaging (BAGI) for mobile, long-range methane detection in locating natural-gas leaks. The technique involves laser radiation of the field of view of an imaging device at the frequency of an absorption line of the gas species to be detected.

Bucin and Cuciuat (4S) proposed an electrical conductivity detector for monitoring the presence of propane in petroleum gas exiting from an adsorption separation unit; use of this device could prevent the loss of propane. Eiceman et al. (7S) quantified the polycyclic aromatic hydrocarbon content in natural-gas samples and residues on the inner wall of natural-gas pipelines using GC-MS and gas chromatography. Of interest was the movement of toxic or carcinogenic compounds in gas-distribution lines. The methane numbers of natural gases were calculated by Vasil'ev and Renkov (27S) to correlate with their antidetonating properties when used in engines running on natural gas. Radon in natural gas was measured by Wojcik (28S) and by Cosma et al. (5S). Wollanke (29S) reported on the nitrogen isotope distribution in the natural gases of southern West Germany, while Koncz (12S) interpreted the carbon isotope ratios of 79 natural gases of Hungary. Hut et al. (11S) separated nitrogen and methane from natural gas by gas chromatography for ^2H , ^{13}C , and ^{15}N determination by mass spectrometry.

The analyses of 276 natural gas samples in the United States were collected by Moore and Hamak (15S), while Hertweck and Fox (9S) published the analyses of natural-gas samples in the United States, Canada, and Thailand. The chemical compositions of natural gases in Japan were reported by Urabe et al. (26S) and Yonetani (30S). Lubas (13S) correlated the concentration of mercury in natural gas to natural-gas compositions in Polish lowlands.

STANDARDS

Cowper and Wallis (3T) presented recommendations for improving the accuracy and precision of natural-gas analysis by gas chromatography. They suggested a single injection with a flame ionization detector linked to a thermal conductivity detector. Air contamination should be quantified and a high-quality, gravimetrically prepared, calibration gas should closely approximate the sample. Koning and van Rossum (6T) discussed preparation methods for calibration gas mixtures used in the quality control and custody transfer of natural gas. Improved accuracy in the gas chromatographic analysis of fuel gases was ensured by Filo (4T) by using two standards of higher and lower concentration than the concentration of the analyzed compound in the fuel-gas sample. The need for

international standardization in the natural-gas industry was emphasized by Bijl and van Rossum (2T).

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 (5T) include the following natural-gas analyses: 6975, Natural Gas—Determination of Hydrocarbons from Butane (C_4) to Hexadecane (C_{16})—Gas Chromatographic Method; 8158, Gas Analysis—Evaluation of the Performance Characteristics of Gas Analyzers.

The ASTM standards (1T) that have been adopted or revised during this review period include the following: D1070-85, Relative Density (Specific Gravity) of Gaseous Fuels; D1142-86, Water Vapor Content of Gaseous Fuels by Measurement of Dew-Point Temperature; D1267-84, Vapor Pressure of Liquefied Petroleum (LP) Gases (LP-Gas Method); D1835-86, Specification for Liquefied Petroleum (LP) Gases; D1837-86, Volatility of Liquefied Petroleum (LP) Gases; D1838-84, Copper Strip Corrosion by Liquefied Petroleum (LP) Gases; D2420-86, Hydrogen Sulfide in Liquefied Petroleum (LP) Gases (Lead Acetate Method); D4150-84, Terminology Related to Gaseous Fuels; and D4468-85, Total Sulfur in Gaseous Fuels by Hydrogenolysis and Rateometric Colorimetry.

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