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Crude Oil and Shale Oil

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This year's review on crude oil and shale oil has been prepared by classifying the references into the following main headings: Hydrocarbon Identification and Characterization, Trace Element Determination, Physical and Thermodynamic Properties, Viscosity, and Miscellaneous Topics. In the two-year review period, the references on shale oils were considerably less in number than those dealing with crude oils. Several new analytical methodologies and applications were reported for hydrocarbon characterization and trace element determination of crude oils and shale oils. Also included in this review are nine U.S., Canadian, British and European patents.

HYDROCARBON IDENTIFICATION AND CHARACTERIZATION

Lundanes and Greibrokk presented a review with 109 references of the analytical techniques (FIA, GC, GC/MS, HPLC, SFC, TLC, NMR, MS, IR) employed for compound class determination, preparative fractionation, and subgroup determination in fuels, heavy fractions, and crude oils (B1). For determining the aromatic content and aromaticity of oil sand processing streams, Crickmore et al. compared the results from several spectroscopic and chromatographic analytical techniques (B2).

Gas Chromatography (GC). Philp presented a review with 68 references of the application of high-temperature gas chromatography (HTGC) to the analysis of fossil fuel samples (B3). Yancey et al. outlined the importance and advantages of a quality control scheme for achieving reproducible capillary GC characterization of crude oils and petroleum condensates (B4). A procedure for chromatographic analysis of hydrocarbons, involving the irradiation and absorbance of ultraviolet light, was described in a Canadian patent (B5).

By identifying a number of alkylcyclobenzenes in crude oils by GC retention behavior and mass spectra, Alexander et al. concluded that the crude oils with abundant methyl- and dimethylcyclohexylbenzenes also have abundant alkylbiphenyls (B6). Ioppolo et al. described a procedure for the identification and analysis of phenols in crude oils, which involves extraction with alkaline aqueous methanol followed by a back extraction for the removal of carboxylic acids (B7). The phenols were analyzed by capillary GC-FID and GC/MS.

Budzinski et al. determined the GC retention index of a smectic liquid crystalline phase for 30 C₂-phenanthrenes and 33 trimethylphenanthrenes (B8). The retention behavior of alkylated phenanthrenes was found to be related to molecular shape considerations, e.g., length-to-breadth ratio, dihedral angle, and substitution pattern.

By studying the GC retention behavior and mass spectra, Singh et al. identified norcadalenes and related compounds in sedimentary organic matter (B9). Zhang et al. used on-line GC and isotopic MS analyses of light petroleum hydrocarbons to obtain geochemical information for the Ordos Basin (B10). The deposits in petroleum production pipelines were analyzed using high-temperature high-resolution GC (B11).

Douglas et al. provided a methodology especially suited for measuring trace levels of hydrocarbons, such as in complex environmental matrixes (B12). The procedure utilizes GC-FID

for alkane and total oil analysis combined with GC/MS for polynuclear aromatic hydrocarbon analysis. Schaps et al. described an experimental program for biomarker analysis by GC/MS and data processing (B13).

Liquid Chromatography (LC). Kelly and Bartle presented a review with 25 references of the use of combined LC/GC for the analysis of petroleum fuels, oils, tars, and engine exhaust particulate extracts (B14). Shadle et al. used liquid chromatography to separate shale oils into class types as well as in product characterization (B15). The polar compounds from the 650–1050 °F distillate of an offshore Californian crude oil were subjected to on-line LC/MS analysis to determine the elemental composition and number of exchangeable hydrogen atoms (B16). Carbognani developed a fast LC method for the group-type separation of low-olefinic petroleum middle distillates; the method may find applications in fingerprinting of refinery cuts and upgrading process streams (B17). For separating certain isomers of alkylnaphthalene and alkylphenanthrene from a crude oil fraction, Ellis et al. described a liquid chromatography technique using dealuminated mordenite molecular sieves (B18).

High-Performance Liquid Chromatography (HPLC). McDonald and Kennicutt fractionated oils into aliphatic and aromatic components by HPLC for the quantitative determination of selected biomarkers by GC/MS with selected ion monitoring (B19). Ali and Nofal reported the results of HPLC measurements on the asphaltene, saturated, aromatic, and polar fractions of four Saudi Arabian crude oils with the API gravity of 28–38 (B20).

Akhlaq and Goetze applied reversed-phase HPLC on five fractions of several crude oil samples to obtain information on molecular weights of heteroaromatic compounds and chain lengths (B21). A British patent described a portable apparatus for hydrocarbon analysis by gel permeation chromatography (B22).

Atomic Emission Detection Spectroscopy (AED-AES). The details of an analytical scheme for spectral information and physical properties of crude oils were given in a U.S. patent (B23). Botto provided a discussion of various applications of the ultrasonic nebulization (USN) technique in analyzing petroleum and petrochemicals by inductively coupled plasma (ICP) AES (B24). The advantages of the USN were reported to be high efficiency, sensitivity and desolvation capability.

Mass Spectrometry (MS). Zhou and Tao reported a high-resolution MS method for the determination of petroleum acids in a Chinese crude oil and distillate (B25). The method involves conversion of carboxylic acids to hydrocarbons which are analyzed by high-resolution MS. Hsu et al. demonstrated ultrahigh resolution capabilities of the Fourier transform ICR-MS technique for accurate mass measurements on complex mixtures (B26).

Supercritical Fluid Chromatography (SFC) and Supercritical Fluid Extraction (SFE). Levy presented a review with 52 references on the application of SFE and SFC for sample preparation and analysis in numerous areas of the petroleum industry (B27). Levy et al. highlighted the use of a cryogenically cooled adsorbent trap for capturing the extracted analytes following supercritical fluid extraction to improve their collection and recovery (B28). Peaden presented a review with 57 references

of the use of GC and SFC for simulated distillation and element-specific detection coupled with these techniques (B29). The details and features of a SFC instrument, capable of providing accurate and reproducible analysis, were described by Kornfeld (B30).

Other Analytical Techniques. Sim et al. discussed the application of a multidimensional chromatographic MS technique to the analysis of complex mixtures of polycyclic aromatic hydrocarbons (B31). Comparative performances of molecular sieve (type 5A) and urea adduction were studied in terms of the range and quantitative yield of *n*-paraffins extracted from two Nigerian crude oils (B32).

By using multivariate circular profiles to screen several parameters for differentiating crude oils by their geologic origin, Lai et al. recommended the selected parameters to be photoacoustic spectroscopy (PAS) and HPLC for unsaturated, aromatic, and polar compounds (B33). Olajire and Oderinde discussed the use of IR spectroscopy for the characterization of crude oils, middle distillates, heavy oils, and residues from various Nigerian locations (B34). Hasan et al. separated a Pakistani waxy crude oil into several fractions and characterized each fraction by FT-IR and ¹H NMR spectroscopy (B35).

Blanco et al. tested and compared the extraction efficiencies of ultrasonic and Soxhlet processes for a Spanish oil shale sample (B36). The low-temperature ultrasonic process was reported to be quick and effective, which did not change the biomarker ratios. A method for determining the concentrations of low molecular weight components in crude oils by use of a dilution stripping technique was described in a U.S. patent (B37).

Mullins et al. reported the location of the electronic adsorption edge of more than 20 crude oil and asphaltene samples to vary over a wide range, from near-infrared for heavy oils and asphaltenes to near-ultraviolet for gas condensates (B38). The electronic adsorption edge for crude oil samples was characteristic of the Urbach tail, which was shown to be a direct measure of the population distribution of chromophores.

Hsu and Qian pointed out the advantages of CS₂ charge exchange (CS₂/CE) over low-voltage electron-impact ionization (LV-EI) in hydrocarbon characterization (B39). It was shown that high and uniform sensitivities in CS₂/CE over LV-EI facilitates high-resolution accurate mass measurements. Zhu and Mullins determined the temperature dependence of fluorescence for eight crude oils at 20 to 200 °C and for related compounds at -80 to 200 °C (B40).

TRACE ELEMENT DETERMINATION

Trace Metals. Lobinski and Adams provided a review with 28 references of the potential and applications of GC, microwave-induced plasma (MIP), and AES techniques for the analysis of organometallic and organometalloid compounds (B41). McDaniel et al. reviewed (78 references) techniques involving radionuclide accelerator mass spectrometry (AMS), which has several areas of applications including crude oils for high-sensitivity trace element determination (B42). Filby and Olsen discussed the advantages and disadvantages of instrumental neutron activation analysis (INAA) and ICPMS methods for trace elements in crude oils, oil fractions, and source rock bitumens (B43).

Al-Swaidan achieved detection limits of 2.52 µg/g for vanadium and 0.30 µg/g for nickel in petroleum samples by using a combination of a microemulsion technique, a standard addition

method, and ICPMS (B44). A microemulsion procedure was also described for sample pretreatment in determining trace element analysis by ICPMS; the technique proved to be better than other methods used on Saudi Arabian crude oil samples (B45). In another study, Al-Swaidan used ICPMS for the determination of lead and nickel in Saudi Arabian crude oils, using a MIBK/HNO₃ pretreatment (B46). It was also reported that the addition of 8-hydroxyquinoline improved the extraction efficiency of vanadium and nickel in petroleum samples (B47).

Murillo and Chirinos described a rapid procedure for determining the nickel, vanadium and sulfur contents by ICP-AES on samples prepared by emulsifying crude oil in water and compared the results with those obtained using digestion methods (B48). Borszeki et al. reported a quick sample preparation method for the determination of sulfur and trace metals by ICP emission spectrometry using emulsions (B49). Zeng and Uden investigated sample cleanup procedures and operating variables in the use of high-temperature GC-AED for the detection of vanadyl, nickel, and iron porphyrins in several crude oils (B50, B51). Zeng et al. described the use of an element-selective GC detection by microwave-induced plasma AED for a variety of compounds containing metals, nonmetals, and metalloids (B52). Nali et al. developed a separation step for concentrating nickel and vanadium complexes in characterizing the organometallic species in heavy crude oil fractions by preparative GPC and reverse phase (RP) HPLC combined with graphite furnace atomic absorption (GFAA) (B53).

Barshick et al. extended a previously reported method for trace metal analysis on solution residues by glow discharge mass spectrometry (GDMS) (B54). Bagur et al. described a flame atomic absorption spectrometry (AAS) method for the determination of low concentrations of vanadium in petroleum crudes (B55). Olajire and Oderinde used energy-dispersive X-ray fluorescence spectroscopy to determine Fe, Hg, V, Cd, Pb, Mn, Ni, Zn, Cu, As, and Se in Nigerian crude oils and heavy residues (B56). An attempt was also made to establish the distribution pattern of V and Ni in the samples tested.

Ojeda et al. described a technique for determining vanadium, iron, nickel, and sulfur in crude oils by total-reflection X-ray fluorescence (TXRF) (B57). The detection limits were 20 ppm for S, 0.6 ppm for V, 0.1 ppm for Fe, and 0.4 ppm for Ni; the reproducibility was reported to be 2–6%. Udoh et al. tested *p*-xylenesulfonic acid as an ashing agent in the determination of Ca, Cu, Fe, Mg, Mn, Na, Ni, and Zn in petroleum crude (B58). It was noted that the simultaneous ignition of an oil/acid (boiling) mixture prevented the volatilization losses of some trace elements, while eliminating the coke residue and reducing the ashing time.

Xu and Lasage separated nickel and vanadyl petroporphyrins in oil samples by use of normal-phase HPLC (B59). The procedure was described to be simple and efficient for the fingerprinting of such compounds. Rankin and Czernuszewicz utilized the sensitivity and selectivity of resonance Raman (RR) scattering to establish the structure marker frequencies for petroporphyrins in crude oils and tar balls (B60). They showed that the significant differences in RR spectra reported for nickel and vanadyl etioporphyrins I and III may be used for identifying various isomers in the two metalloporphyrin families.

Ali et al. presented the details of a method for analyzing nickel and vanadium porphyrins in Arabian heavy crude oils (B61). The method involves extraction and separation of porphyrins (by

adsorption chromatography and HPLC) and their characterization by UV/visible and MS techniques. From their study of a shale oil fraction, Wood and Bonham reported that plasma desorption mass spectrometry (PDMS) is an alternative MS method for analyzing mixtures of metalloporphyrins (B62). Carbon number distributions and the recognition of both etiometalloporphyrin and DPEP metalloporphyrin analogs are possible by obtaining PDMS spectra in the reflector mode.

Reid et al. reported the elemental analysis of crude oils and their combustion products from eight Kuwaiti fields (B63). The analytical methods used were proton-induced X-ray and X-ray fluorescence techniques. Raoof and Al-Amoodi used energy dispersive X-ray fluorescence spectrometry for determining vanadium, nickel, and sulfur contents in Saudi Arabian crude oils (B64). Hwang et al. described the synthesis and spectroscopic/EPR characterization of a vanadyl complex with acetone Schiff base (B65). The information provided may be useful in characterizing aqueous pyridine fractions of certain heavy oil asphaltenes.

Yeh et al. synthesized a chelating resin with dithiocarbamate functional groups and reported optimum process conditions for the formation of metal complexes (B66). A method of extracting iron from crude oils or other liquid hydrocarbon streams was described in a Canadian patent (B67).

Sulfur Compounds. By characterizing five crude oils with varying sulfur contents (0.1–4.7%) for organically bound sulfur, Damste et al. identified major sulfur compounds in the aromatic, asphaltene, and polar fractions using GC/MS and flash pyrolysis-GC/MS (B68). The polar fractions were desulfurized with Raney Ni, with the produced hydrocarbons analyzed by GC/MS. Adam et al. discussed the molecular structure and importance of a hexane-soluble nonpolar macromolecular fraction (NPMF) which consists of low molecular weight hydrocarbon subunits cross-linked with sulfide bridges (B69). The NPMF is highly aliphatic with a molecular weight of several thousand mass units. Nuzzi evaluated a differential pulse polarography technique for studying sulfur–sulfur and carbon–sulfur bond scission in the organic sulfur compounds found in crude oils (B70).

Kelly et al. reported good reproducibilities in the total sulfur content for 13 petroleum samples by an isotope dilution thermal ionization MS technique (B71). Nishioka and Tomich developed a nonreactive ligand-exchange chromatographic method for the isolation of aliphatic sulfur compounds in crude oils followed by their analysis using combined GC/MS (B72). The method is based on the observation that, due to their higher nucleophilicity, the aliphatic sulfur compounds form stronger ligands with CuCl_2 than the aromatic sulfur compounds. Green et al. presented a semiquantitative method for the structural characterization of sulfur compounds in petroleum via their conversion to corresponding methylsulfonium salts followed by ^{13}C NMR analysis (B73).

Shearer and Skelton investigated the use of SFC with flameless sulfur chemiluminescence detection for sulfur compounds in petroleum products (B74). They found the detection system to exhibit good precision, linearity, selectivity and sensitivity, and nearly linear equimolar response. Murillo et al. described a procedure for rapid determination of sulfur in crude oils by a Parr bomb digestion method followed by the ICP-AES analysis (B75). The method was verified against the results using X-ray fluorescence on Venezuelan crude oils and a NIST standard.

Bark and Sumardi described a rapid direct injection enthalpimetry method for the determination of thiol S and H_2S over the concentration range of 0–100 mM (B76). Long et al. used HPLC, IR spectroscopy, and GC/MS for the separation and structural determination of petroleum sulfoxides (B77).

Nitrogen Compounds. Lancas and Barbirato described a method for selectively isolating neutral nitrogen-containing fractions of crude oils by use of (i) column liquid chromatography using silica gel modified with titanium(IV) oxide and (ii) preparative liquid chromatography using thermally treated silica gel as the stationary phase (B78, B79).

A chromatography-based isolation method, using an ion-exchange resin with sulfonic acid active centers, was developed for the isolation of oxygen- and nitrogen-containing compounds in Kirkuk crude oil, followed by their characterization by IR and ^1H and ^{13}C NMR (B80–B82). Nwadinigwe and Maduka reported the results for the total, basic, and nonbasic nitrogen contents of 28 Nigerian wellhead crude oils and their fractions (B83). The nitrogen content was noted to vary with the boiling range of the fractions.

PHYSICAL AND THERMODYNAMIC PROPERTIES

Ashcroft et al. measured and modeled the volumetric behavior of 7 European, South American, and African crude oils combined with 10 light hydrocarbons or hydrocarbon mixtures (B84). For these nonideal systems, depending on the nature of the mixture constituents, the excess volume may be positive or negative. A calculation method for characterizing petroleum fractions as pseudocomponents was described, which requires boiling point distribution curve and density information for estimating Watson's characterization factor (B85). Tu developed a method for calculating the normal freezing point of hydrocarbons from structural information by matching the data for 307 hydrocarbons with an average deviation of 7.4% (B86).

Riazi and Mansoori developed an equation of state model for the density of hydrocarbons ($\text{C}_1\text{--C}_{40}$) and their mixtures, which can be used at temperatures up to 1000 K and pressures up to 70 MPa (B87). The effect of pressure (up to 1 GPa) on the isothermal compressibility of two crude oils at room temperature was measured and correlated (B88). The data indicated a strong dependence of crude oil compressibility on the viscosity and composition. Kutcherov et al. measured the glass transition temperature of Usinsk crude oil at 200–400 K at pressures up to 1 GPa by the transient hot-wire technique (B89). It was found that the higher the pressure, the narrower the glass transition temperature. Deqing et al. measured and correlated the critical temperature, pressure, and volume of 102 fractions from five Chinese crude oils (B90). The critical properties correlations were compared to the predictions obtained from a group contribution method.

Gad developed a computer program, based on an adaptation of literature data, for estimating physical properties of crude oils (B91). Baskin and Jones provided empirical correlations for estimating the API gravity, prior to drill-stem testing, primarily from sulfur content and pyrolysis data of the bitumen extracted from reservoir rock samples of Monterey formation in California Offshore (B92). A previously published correlation by Dokla and Osman for the pressure/volume/temperature (PVT) relationship of crude oils was reported to be inadequate due to the use of limited data (B93).

An experimental study by Verrall et al. suggested that measuring the sound velocity may provide an accurate on-line monitoring of viscosity, density, diluent-to-oil ratio (<50 wt % toluene as diluent), and water-in-oil ratio (<10 wt % added water) at pipeline conditions (B94). Methods based on dual-energy γ -ray transmission (DUET) techniques were reported for determining the liquid velocities and the volume fraction information for oil/water/gas mixtures (B95, B96).

A spectrophotometric method for screening and determining the optimum dosage of antifouling agents, used for dispersing asphaltenes in crude oils, was outlined in a U.S. patent (B97). Kok and Okandan used differential scanning calorimetry (DSC) to determine changes in the calorific value in mixtures of lignite and Turkish crude oils (B98).

Waxy (Paraffinic) Crude Oils. Khan et al. determined the wax appearance temperature of three petroleum waxes dissolved in various solvents under varying thermal conditions (B99). Dilawar et al. separated waxes from Bombay High (India) crude oil to study the effect of a pour point depressant additive on their structural change by X-ray diffractometry (B100). Khan et al. described an accurate method for determining the wax appearance temperature to $\pm 1^\circ\text{C}$ (B101). It was also reported that the pour point of waxy crude oils can be determined from a flow characteristic called "restart" pressure.

A photoanalysis technique was used to study wax crystal modification due to the addition of certain flow improvers during crystal growth at low temperatures in Egyptian crude oils (B102). An apparatus and procedure for determining the wax appearance point and wax content of petroleum products was described in a British patent (B103).

Water/Oil Emulsions and Suspensions. Upton reviewed the monitoring and standardized testing of sediment and water content of crude oils (B104). Poynter and Barrios discussed the coulometric Karl Fischer titration technique for water determination in crude oils and petroleum feedstocks (B105). The topics covered in their discussion include the history, ASTM tests, coulometric titration, and reactions involved.

Chen et al. investigated the effect of an ac electrical field on the structure of a water-in-oil emulsion experimentally and also theoretically using a molecular dynamics simulation (B106). A rigid interfacial film, resulting from adsorbed crude oil components (e.g., asphaltenes), was concluded to be an important factor for preventing the coalescence of water droplets in the electrical field. Aggarwal and Johnston developed a technique, based on the use of X-band microwaves (10.5 GHz) together with the salinity, temperature, and configuration information, for determining water content in petroleum pipelines (B107).

An apparatus for measuring the water content in crude oils containing entrained gases, based on the difference between incident and transmitted microwave power through the sample, was described in a U.S. patent (B108). In a European patent, an apparatus that utilizes the measurement of transmittance of a narrow band of infrared (IR) was described for determining the dissolved water content of hydrocarbons (B109).

VISCOSITY

Orbey and Sandler presented models for calculating the viscosity of hydrocarbons and their mixtures, including crude oils, over a wide range of temperatures and pressures (B110). In many cases, the model is predictive, requiring only the normal boiling

point of the constituents as input data.

Roenningsen presented viscosity/temperature/molecular weight correlations for North Sea petroleum liquids (B111). In addition, an empirical relationship between viscosity and surface tension was also proposed. Ali evaluated two viscosity estimation methods: a residual viscosity correlation and another based on the extended corresponding states principle (B112). The residual viscosity correlation was found to be very sensitive to the liquid-phase density, which is a required input property. van der Walt et al. illustrated the use and "training" of a neural net (a three-layered perceptron) for correlating the viscosity of crude oils and fractions (B113). Labedi presented correlations for the viscosity of Libyan petroleum products in terms of stock tank oil gravity (density), pressure, and temperature (B114).

Miadonye et al. presented a correlation for the effect of temperature on the kinematic viscosity of crude oil fractions, which requires one measurement on each fraction at 100°F (B115). Similarly, Singh et al. correlated the effects of temperature and pressure on the kinematic viscosity of Middle East crude oils and their mixtures, which requires one measurement at 30°C on each sample (B116). In a correspondence, the correlation by Singh et al. (B116) was shown to give erroneous results and to be limited in its applicability to only those crude oil mixtures that had been used in obtaining the correlation (B117).

Waxy (Paraffinic) Crude Oils. Whereas most crude oils show Newtonian behavior under commonly encountered conditions, waxy crude oils show complex non-Newtonian characteristics which are dependent on several parameters. Wardhaugh and Boger discussed the yielding behavior of statically cooled waxy crude oils (B118). They described the yielding process to have three distinct characteristics: a solid behavior, a slow (creep) deformation, and a sudden failure resembling the brittle or ductile failure of solids. Means of obtaining reproducible yield stress and fracture stress data were also discussed. On the basis of rheological measurements with a rotational viscometer, Al-Fariss et al. developed a correlation for the viscosity of waxy crude oils as a function of shear rate, temperature ($9\text{--}24^\circ\text{C}$) and wax content ($2\text{--}8\text{ wt } \%$) (B119).

MISCELLANEOUS TOPICS

Shadle et al. carried out high-temperature, short-contact-time pyrolysis of oil shales to investigate the reaction mechanisms of kerogen decomposition, and the results were compared with those from low-temperature, slow-heating (conventional retorting) processes (B120). It was concluded that, as a result of different reaction mechanisms, the high conversion of kerogen in the rapid pyrolysis process produces a low-quality product which would require upgrading before utilization.

Premuzic et al. examined the reactions induced in petroleum by natural or added acidophilic and pressure-adapted thermophilic or thermally adapted microorganisms (B121). The results may find applications in microbial-enhanced oil recovery where chemical changes proceed with oil recovery.

A multiphase mass flowmeter, based on the direct relationship between density and dielectric constant of hydrocarbon mixtures, was described in a U.S. patent (B122).

The use of microwave signals was described in a British patent on an apparatus for determining the gas and water contents in crude oil pipelines (B123).

Anil K. Mehrotra is a professor in the Department of Chemical and Petroleum Engineering at the University of Calgary. His current research interests include transport and thermophysical properties and phase equilibria of heavy oils, bitumens and paraffinic crude oils, asphaltene/wax deposition kinetics, and soil remediation. He is a registered professional engineer with APEGGA, and a member of CIC, CSChE, and AIChE. He received a B.E. in chemical engineering from B.I.T.S. (Pilani, India, 1972), an M.Eng. in environmental engineering from A.I.T. (Bangkok, Thailand, 1975), and a Ph.D. in chemical engineering from the University of Calgary (Calgary, Canada, 1980).

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