On the Chromatographic Liberation of Aromatic Hydrocarbons From Their Mixture With Organosulphur Compounds 20-114-4-34/63

ASSOCIATION: Department of Chemistry of the Bashkir Branch of the AS USSR (Otdel khimii Bashkirskogo filiala Akademii nauk SSSR)

PRESENTED: December 25, 1956 by B. A. Kazanskiy, Member, Academy of Sciences, USSR

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OBOLENTSEV, R.D.

11(4) pr,4,78,9,12,13 PHASE I BOOK EXPLOITATION SOV/1319

Akademiya nauk SSSR. Bashkirskiy filial

Khimiya sera-organicheskikh soyedineniy, soderzhashchikhsya v neftyakh i nefteproduktakh; materialy II nauchnoy sessii (Chemistry of Sulfur-Organic Compounds Contained in Petroleum Products; Papers of the 2nd Scientific Session) v. 1. Ufa, Izd. Bashkirskogo filiala AN SSSR, 1958. 228 p. 1,500 copies printed.

Ed.: Sudarkina, K.I.; Editorial Board: Ayvazov, B.R., Mashkina, A.V., Obolentsev, R.D. (Resp. Ed.), Rozhdestvenskiy, V.P., and Shanin, L,L.; Tech. Ed.: Makhimov, R. Sh.

PURPOSE: This book is intended for petroleum specialists of scientific research establishments, educational institutions, and petroleum refining plants.

COVERAGE: This collection is the first of a multivolume publication on the results of scientific research work carried out in the Soviet Union on the chemistry and technology of sulfur- and nitrogen-organic compounds during the period 1954-1955; and according to a coordinated research project outlined in 1956 by the sponcering

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agency (Bashkir Branch of the Academy of Sciences USSR). Along with the 22 reports published herein, abridged versions of questions, answers and discussions are given wherever the editors deem it expedient.

TABLE OF CONTENTS:

Sulfur-organic compounds were separated from kerosene fractions of petroleum and physical constants (including molecular formulas, refractive indices, etc.) were determined corresponding to mono-, bi- and tricyclic sulfides. Experimental data on the fractional distillation of these compounds (which vaporized at 209-210° C) compared with known data identified them as 3-butylthiophanes [tetrahydro 3-butylthiophenes]. A.D. Biktasheva and N.S. Lyubopytova carried out the spectrographic analyses.

Ivanova, N.M., Ch. Kh. Mirkhaydarova, and Ya. I. Nel'kenbaum (Ishimbayskiy neftepererabatyvayushchiy zavod--Ishimbay Oil Refining Plant)
Installation for Chromatographic Separation of Sulfur-Containing Compounds From Petroleum Distillates 29

Illustrations, schematic diagrams of apparatus and a table of data are given for the chromatographic analysis of the sulfur content of Ishimbay petroleum after pyrolysis.

Gorskaya, N.G. (Novo-Ufimskiy neftepererabatyvayushchiy zavod -- New Oil Refining Plant at Ufa) On the Problem of Constructing Larger Chromatographic Installations for Separating Concentrates of Sulfur-Organic Compounds From Petroleum Products 38

Ten tons of petroleum material with a sulfur content of 0.15 percent was processed by the chromatographic method with a separation yield of 70 percent concentration of sulfur-organic compounds amounting to 80 liters after 9 months (approximately) of continuous operation.

Skripnik, Ye. I. (Kuybyshevskiy industrial'nyy institut -- Kuybyshev Industrial Institute). Thermal Stability of Sulfur-Organic Compounds of Sulfur-bearing Petroleum From Kuybyshevskaya Oblast' 43

According to the author, Kuybyshevskaya oblast' ranks third in extracting and refining petroleum in the Soviet Union during the Sixth Five Year Plan. Separation of sulfur-organic compounds from the highly sulfurous petroleum of this region (3-4.5 percent; tar - up to 22 percent; and in asphalt-tar - up to 100 percent S) was accomplished by two methods: 1) decomposition of complex sulfur-organic compounds 2) reaction of elementary sulfur with hydrocarbons. The first process took place at 350° C and gave maximum sulfur-compound formation at 400° C and higher. The second process took place at 180 - 220° C with a high yield of sulfur compounds. Tabular results of these processes are given.

Obolentsev, R.D. and B.V. Ayvazov. Thermal Stability of Sulfur-Organic Compounds Contained in Petroleum From the Tuymazy Oilfield 51

Two types of petroleum (from Carboniferous and Devonian deposits) were heated (150 - 300° C) and graphs, tables and equations are given for the separation of petroleum compounds with respect to heating time and temperature.

Zakharochkin, L.D., and S.T. Meshcheryakov, (Gosodarstvennyy nauchnoissledovatel'skiy i proyektnyy institut neftyanogo mashinostroyeniya--State Scientific Research and Planning Institute for Petroleum Machinery Building). On the Problem of Evaluating the Corrosive Properties of Sulfurous Petroleum. 65

Oil from various horizons (Devonian, Carboniferous, Upper Permian, etc.) of Ural-Volga deposits was tested for free sulfur content, yield of H_2S on distillation, and speed of corrosion of steel (the latter two factors were determined at temperatures up to 350° C). The purpose of the investigation was to establish criteria for selecting, storing, transporting and refining sulfurous petroleum from different fields. N.V. Tokareva, O.V. Kalinina and G.G. Zhukova assisted in the experimental work.

Chertkov, Ya. B., and V.N. Zrelov, Nauchno-issledovatel'skiy institut goryuche-smazochnykh materialov--Scientific Research Institute for Fuel and Lubricating Materials). Activity of Sulfur-Organic Compounds in Relationship to the Metal

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of the Fuel System of Gas-Turbine Engines

Various fuels from the ligroin-kerosene fractions of petroleum, products of both direct distillation and thermal cracking, with an average content of sulfur (0.12 - 0.94 percent), mercaptan (0.004 - 0.060 percent and elementary sulfur (0.001 - 0.010 percent), were investigated for corrosive, resin- and residue-forming properties in relationship to copper, bronze, cadmium, zinc and chromium-steel alloys with various surface finishes (nitrided, casehardened, etc.). Illustrations of laboratory apparatus, graphs of the corrosive effects of elementary sulfur and aliphatic and aromatic mercaptans, and tables showing the content of these substances in fuels are given.

Tayts, S.Z. (Institut organicheskoy khimii AN SSSR--Instutute of Organic Chemistry, AS USSR). Synthesis of Compounds of the Aliphatic Series on the Basis of Thiophene and Its Homologs 80

The author states that tar from Povolzh'ye(Volga region) shales and sulfurous petroleum may serve as inexhaustible sources of thiophene and its homologs. Reaction diagrams are given for the synthesis of aliphatic compounds from this material, which yielded C_4-hydrocarbons, higher alcohols, glycols, acids, oxy-acids, α, β, γ- and other amino acids, amino dicarboxylic acids, tertiary amines, amino alcohols, simple esters, etc.

Thirty different sulfur-organic compounds were synthesized to facilitate the investigation of the negative effects of these compounds in the extraction and refining of petroleum and to work out rational means for petroleum desulfurization. Synthesis reaction diagrams and physical constants of the synthesized compounds are given.

This investigation is based on the cryoscopic method. From an initial approximation, $N_2 = \frac{\Delta H_{fu} \Delta T}{RT_o^2}$ (where: N_2 -molar amount of admixture with respect to a decrease in freezing point; $\Delta T = T_o - T_1$, ^oK, T_o - freezing point of a pure substance, ^oK; T_1 - freezing point

of the sample substance, ^{o}k; H_{fu} - heat of fusion of a pure substance at T_o, cal/mol; R- gas constant, cal/mol degree [^{o}C], graphs and tables of freezing point, purity, and cryoscopic constants are given. Schematic drawings of laboratory set-ups are included.

by a 1 percent solution of hydrogen peroxide. The sulfuric acid formed is titrated with a 0.02N solution of sodium hydroxide in the presence of a mixed indicator (methyl red-methylene blue). Resultant data is tabulated and compared with data obtained by other methods.

In contrast to the usual polarographic method (where curves of the dependency of the current "I", passing through the solution, on the applied electromotive force "E" are derived), this method investigates the dependency of the speed of current change

$\frac{dI}{dE}$ on the applied e. m. f. "E." A differential polarogram of nitrate salts of lead and thallium showed two distinct maxima which corresponded to the presence of two cations in the solution.

It is noted that a high content of sulfurous mercaptans in fuels causes the corrosion of fuel equipment. A method invented by B.G. Adams was used to determine the content of sulfurous mercaptans in mixtures of kerosene and synthetic mercaptans, and straight-run fuels. This method is based on the capacity of mercaptans to form copper mercaptides by reacting with an ammonium solution of copper sulfate. Results are tabulated.

A process of "automatic hydrogen purification" (avtogidroochistka) is described which consists in the use of hydrogen separated during the dehydrogenation of naphthene hydrocarbons, as proposed by F.W.B. Porter (Refs 1, 2). Desulfurization of kerosene distillates with initial sulfur content up to 0.8 percent was 90-95 percent after boiling at temperatures ranging from 140 to 300°C for 1000 hours; whereas, desulfurization of gas oil fractions of ~ 1 percent sulfur content was 60-80 percent after 200 hours at 200-350°C.

A coarse concentrate of finely stamped ore is brought into contact with sulfurous gasoline vapors at 450-550° C. Mineral ores containing compounds of metals show catalytic properties. With the rupture of C-C or C-S and C-H bonds, these minerals (depending upon their properties) are reduced from sulfides and are covered with coke films. These changes may be exploited for flotation or other methods of enriching ore. Catalytic cracking takes place simultaneously. (Data are tabulated and other facets of the process are discussed).

Stankevich, B. Ye. (Bashkirskiy nauchno-issledovatiel'skiy institut neftyanoy promyshlennosti -- translated in title). Efforts of the Bashkir Scientific Research Institute for the Petroleum Industry to Reduce Expenditures for Caustic Reagents 162

Methods are proposed for circumventing the expensive and extremely difficult regeneration of spent caustics: a) blowing through a spent caustic at ~100°C with a mixture of water vapor and compressed air b) electrolytic regeneration -- (in experimental stages), and c) substitution of trisodium phosphate (TSP) for caustic soda. Laboratory tests with an experimental set-up producing 50 liters per hour showed that distillates purified with TSP passed the copper plate tests.

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PHASE I BOOK EXPLOITATION 647

Akademiya nauk SSSR. Institut nefti

Sostav i svoystva vysokomolekulyarnoy chasti nefti; sbornik rabot po izucheniyu sostava i svoystv neftey i nefteproduktov (Composition and Properties of the High Molecular Weight Fraction of Petroleum; Collection of Papers on the Composition and Properties of Crudes and Petroleum Products) Moscow, Izd-vo AN SSSR, 1958. 370 p. 3,500 copies printed.

Resp. Ed.: Sergiyenko, S.R., Professor; Ed. of Publishing House: Nekrasov, A.S.; Tech. Ed.: Pavlovskiy, A.A.; Editorial Board: Topchiyev, A.V., Academician, Kazanskiy, B.A., Academician, Fedorov, V.S., Candidate of Technical Sciences, Kusakov, M.M., Professor, Sergiyenko, S.R., Professor, Plate, A.F., Professor, Nikolayeva, V.G., Candidate of Technical Sciences.

PURPOSE: This collection of articles introduces new material in the field of high molecular weight components of petroleum. It is intended for scientific and engineering personnel.

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COVERAGE: This book is the second collection of articles published by the All-Union Conference on the Composition and Properties of Petroleum Products, held in January 1956 in Moscow. This volume contains papers on the composition and properties of high molecular weight compounds and sulfur compounds in petroleum. Several of the original papers were revised by the authors, other papers were changed by the editing committee. The articles add to the knowledge of the chemical nature and properties of the least studied fraction, that is the high molecular weight fraction of petroleum. Many new experimental data are given on the composition and properties of the high molecular weight fractions of Soviet crudes (lubricating oils, paraffins, resins and asphaltenes, sulfur compounds). Research methods are discussed. Present and future trends in research are explored.

TABLE OF CONTENTS:

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PART I. SOME BASIC PROBLEMS OF MODERN PETROLEUM CHEMISTRY

sulfur compounds found in Soviet crudes. Attention is paid to the recovery of sulfur compounds from high boiling point fractions. There are 7 tables, and 14 references of which 11 are Soviet and 3 English.

Gal'pern, G.D. Physicochemical Properties and Composition of Petroleum Products 36

The author gives a general review of the field of petroleum chemistry. Foreign and Soviet developments are discussed including methods for the determination of the composition and properties of hydrocarbons in multicomponent hydrocarbon liquids. There are 36 references of which 15 are Soviet, 15 English, 2 German, 3 Dutch, and 1 French.

PART II. STUDY OF THE HYDROCARBON COMPONENTS OF OILS

Tilicheyev, M.D., Borovaya, M.S., Buk, N.S., Okinshevich, N.A., Gryoscopic Method for Quantitative Determination of Aromatic Hydrocarbons in Petroleum Oils in Cyclohexane Solution 47

Composition and Properties of the High Molecular (Cont.) 647

This article describes the improved cryoscopic method of Tilicheyev and mentions its application for the first time to control of the clearness of separation of aromatic hydrocarbons from alkanes and cyclanes in the chromatographic separation of lubricating oils into aromatic and saturated components with silica gel. It was determined that various aromatic fractions separated from petroleum oils by chromatography (under conditions chosen by the author) contained 2 to 22 mol. percent of nonsulfonating admixtures. There are 4 tables and 3 Soviet references.

Zherdeva, L.G., Sidlyaronok, F.G. The Structure and Properties of Aromatic Compounds Contained in the High Boiling Petroleum Fractions 54

This article considers the structure and properties of aromatic compounds from two types of eastern petroleum: Tuymazy petroleum (paraffinic, sulfur containing) and Emba petroleum (low paraffin, low sulfur content). It was determined that high molecular weight aromatic compounds separated from sulfur-containing petroleum consist of a mixture of polycyclic (3 - 7 cycles in an average molecule), mostly condensed aromatic hydrocarbons, and sulfur compounds similar in their properties to aromatic hydrocarbons. The calculation of the structural

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group composition from physicochemical constants without hydrogenation shows considerable disagreement with the composition determined on the basis of hydrogenation, and therefore cannot be used for fractions of polycyclic high molecular weight aromatic compounds. There are 10 tables, 1 figure, and 15 references of which 7 are Soviet, and 8 English.

Maumyan, V.Ya, Stepanyan, T.S., Musayev, M.R. Determination of the Hydrocarbon Composition of Oil Fractions 69

In order to explain the relation between quality of oils and hydrocarbon composition the authors studied a number of oils from the Baku region. They came to the conclusion that the adsorption method of analysis is the most objective one and should be recommended for the determination of hydrocarbons in oils. It is sufficient to examine the fraction with viscosity $E_{50} = 7$ (table 15) in order to obtain the characteristics of the entire range of oils of the studied crude. There are 15 tables, and 3 Soviet references.

powder, the MS-20 shows the following group composition (percent by weight): naphthene-paraffin hydrocarbons 70.3: naphthene-aromatic hydrocarbons 27.1; propane soluble tars 0.7; and tarry substances not soluble in propane 1.9. There are 20 tables and 2 English references.

Kaverina, N.I., Losikov, B.V., Fedyantseva, A.A. Adsorption Method for Determining the Fractional Composition of High-Polymeric Lubricating Oil Additives 109

The authors show that chromatographic adsorption can be used as a method for separating polyisobutylene and vinypol, with active carbon or silica gel as adsorbent. The method can be of considerable interest in estimating the quality of high-polymer viscous additives in oils. It can also be used in obtaining polymer fractions with equal molecular weights. There are 5 tables, 1 figure, and 3 references of which 2 are German and 1 English.

PART III. EFFECT OF COMPOSITION ON THE PERFORMANCE OF LUBRICATING OILS

Kuliyev, A.M., Kuliyev, R.Sh., Aliyev, M.I. Effect of the Hydrocarbon Composition on the Physicochemical Properties and Performance of Lubricating Oils 119

A study was made of the narrow oil fractions and commercial oils obtained from various Baku crudes. It was shown that the physicochemical properties and the performance properties of oils are modified by the hydrocarbons composition and structure. The naphthene-paraffin hydrocarbons obtained from various crudes are similar in quality and have very good temperature-viscosity properties but show low oxidation stability. Aromatic hydrocarbons differ in their properties and have a greater effect on the quality of lubricating oils than naphthene-paraffin hydrocarbons. Aromatics and tars inhibit the action of depressants and additives. The article contains 16 tables there are no references.

Kreyn, S.E., Borovaya, M.S. Effect of the Chemical Composition of Petroleum Lubricating Oils on Their Properties 138

This paper is a study of petroleum oils obtained from various Baku crudes. Components were separated by adsorption. The distillates

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were refined by sulfuric acid and solvent processes. The effect of the composition and the hydrocarbon structures on the quality of lubricating oils was determined for several types of oils. The role of quantity and structure of aromatics, naphthene-aromatics, hydrocarbons, resins and sulfur compounds was studied in motor oils as a factor modifying the character of naphthenic-paraffinic hydrocarbons. The type of the crude and the purpose of the lubricating oil determine the refining processes and their extent. There are 23 tables and 9 references of which 5 are Soviet and 4 English.

Vinogradov, G.V., Kreyn, S.E. Chemical Composition and Wear-Resistance Properties of Petroleum Oils 167

Various types of NPF oils (naphthene-paraffin fractions) were studied on friction-test machines in order to establish their wear-resistance properties in relation to their chemical composition. Their wear-resistance properties depend on the amount of aromatic fractions (AF) which are sulfur bearing, in relation to the NPF of variable viscosity and oxidation stability. The chemical composition of oils and individual fractions determines their characteristic behavior in relation to metals. These characteristics vary throughout the entire range of products from distillates through oils to NPF fractions. The article gives 14 figures and 1 table. There are no references.

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troleum products were tested (e.g. kerosene fractions). The performance of lube oils was examined at high surface friction and with various additives (sulfur, phosphorus, chlorine). Oils used were: transformer oils, SU, AK-15. A close study of the NPF (naphthene-paraffin fraction) was made,and their characteristics were determined as modifying the properties of the oils. The NPF from various crudes are different and their sensitivity to additives vary (especially towards organophosphoric wear-resistance additives). There are 2 tables, 2 figures, and 2 Soviet references.

Pavlovskaya, N.T., Vinogradov, G.V., Bezborod'ko, M.D. Wear-Resistance Properties and Oxidizability of the Naphthene-Paraffin Fractions of Viscous and Low-Viscosity Petroleum Oils 198

Since friction tests show the importance of oil composition, in particular of the NPF, a through study was made of this fraction. The NPF of transformer oil and of MS-20 were used in these tests. Results show that it is possible to achieve an exact differentiation of the various naphthene-paraffin fractions obtained from petroleum oils with different viscosity indexes. It was shown that the NPF of low-viscosity oils have a lower oxidation stability. There are 5 figures and 3 Soviet references.

PART IV. THE CHEMICAL NATURE OF SOLID PETROLEUM HYDROCARBONS

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reagent for normal paraffins. Only slight branched paraffins easily form urea complexes. Most of the isoparaffins which were separated from the Groznyy paraffin as urea complexes have slightly branched structures. Hydrocarbons which do not react with urea resemble the cycloparaffins. There are 6 figures, 2 tables, and 39 references of which 8 are Soviet, 24 English and 7 German.

Voznesenskaya, Ye.V., Zherdeva, L.G. Study of Solid Hydrocarbons From Sulfur-Containing Eastern Crudes 218

It was determined that highly refined solid hydrocarbons obtained from a deasphalted Tuymazy crude concentrate (b.p. $>$ 350°) belong to the methane series and are mainly normal paraffins (C_{23} to C_{36}). Solid hydrocarbons with m.p. $>$ 65° contain about 35 percent isomeric methane hydrocarbons. There are 12 figures, 6 tables, and 19 references of which 9 are Soviet and 10 English.

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products which were determined to be hydrocarbons (high paraffin content and low resin content) and nonluminescent reddish-blue products (nitrogen containing compounds). Evidently, the high molecular weight compounds contain aromatic rings and other structures of low thermal stability, which fact can be regarded as confirmation of the low-temperature formation of petroleum. There are 2 tables and 19 references of which 14 are Soviet and 5 English.

PART V. RESINOUS-ASPHALTIC SUBSTANCES. COMPOSITION, PROPERTIES, AND RESEARCH METHODS.

Sergiyenko, S.R., Davydov, B.E. Physical Properties of Petroleum Resinous Substances 245

Resinous substances from the Romashkino crude and from the Gyurgyany crude were taken for this study. The increase of the amount of acid and neutral saponifiable substances is directly proportional to the increase of the amount of hetero-atoms they contain (O,S,N). All resinous substances are characterized by considerable surface activity. They can be separated into fractions of increasing surface activity

with the aid of a series of solvents with increasing dielectric constants. The differentiation of resinous fractions can be improved by the inclusion of cyclohexane as a solvent. The molecular, surface, and polarization characteristics are used for the differentiation of fractions which show similar results in chemical analysis. There are 2 tables, 9 figures, and 7 references of which 5 are Soviet, and 2 English.

Ben'kovskiy, V.G. Certain New Methods for the Separation of Resinous Substances 258

The author proposes the following types of methods for the separation of resinous substances: methods of collodial chemistry and electrochemistry (electrophoresis, electrodialysis, high-voltage electrolysis, thermodiffusion), and the method of molecular compounds (reaction of organic compounds with salts of various elements). There are 2 figures, 1 table, and 14 references of which 5 are Soviet, 8 German, and 1 English.

Velizar'yeva, N.I., Zherdeva, L.G. Physicochemical Study of Asphaltic-Resinous Substances From Eastern Crudes 266

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Propane treatment yields products with a high hydrogen content and high molecular weight. Phenol treatment gives products of lower molecular weight, high density, and high S, N and O content. Resins from Tuymazy and Emba crudes are composed of polycylic compounds containing S, N, and O, with average molecules of 4 - 6 cycles. The average molecule contains not only aromatic cycles, but also considerable amounts of naphthenic (sometimes up to 50%) and paraffinic (40 - 50%) cycles, basically short. There are 7 tables, 3 figures, and 13 references of which 12 are Soviet and 1 German.

Bogdanov, N.F. Plan of a Standard Method for the Study of Mazut 280

The proposed method is not in its definitive form. Changes and additions are expected after the completion of work on problems which still remain unsolved, both in apparatus and methodology. The proposed method consists of two stages. The first stage which is identical for all mazuts determines hydrocarbon composition and physicochemical properties. The second stage is concerned with the separation of technical products for particular applications. These products are characterized by technical, physical, and chemical properties which should fulfill some requirements, therefore this stage is not uniform for all mazuts but is dictated by specific needs. There are 2 tables, 1 figure, and 4 references of which 2 are Soviet and 2 English.

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conveniently used in plant laboratories. It introduces new elements into asphalt analysis: more complete quantitative analysis of organic components, especially of paraffinic acid asphalts (for the first time), determination of carboxylic acids (naphthenic and asphaltous), determination of "oxonium" compounds of resins and asphaltenes as independent from the various groups of asphaltene-sulfonic acids, and the discovery of the hitherto unknown new group of asphaltene-sulfonic acids which are not soluble in acetone. There are 3 tables and 8 references of which 4 are Soviet, 1 German, and 3 English.

PART VI. PETROLEUM SULFUR COMPOUNDS AND METHODS FOR THEIR INVESTIGATION

Luk'yanitsa, V.G., Gal'pern, G.D. Methods for Study of Group Composition of Sulfur Compounds in Petroleum Products 333

The article reviews the existing methods for the determination of sulfur compounds in petroleum products, and enumerates foreign as well as Soviet methods for the analysis of separates classes (free sulfur, hydrogen sulfide, mercaptans, disulfides, sulfides, thiophenes) and for group analysis. There are 3 tables, 1 figure, and 73 references of which 17 are Soviet, 45 English, 5 German, 2 Czech, and 4 French.

Gusinskaya, S.L. The Nature of Sulfur Compounds in Crudes From Southern Uzbekistan 343

It was determined that Southern Uzbekistan crudes have a high sulfur content (3 - 6 percent) and high content of nitrogen compounds (up to 1 percent). Thiophane homologues were detected (methyl-amyl-heptyl) in these crudes. Uch-Kzyl crudes include also thiazoles (methylthiazole). Gasoline and kerosene from these crudes show 2 - 4 percent sulfur There are 5 tables and 21 references of which are 15 Soviet, 3 English and 3 German.

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Chudakova, I.K., Volynskiy, N.P. Determination of Sulfur Content in Heavy Petroleum Products by Double Combustion 352

This is a new method proposed for the double combustion for the determination of sulfur in all types of petroleum products, with the exception of gasoline and low-sulfur kerosene, and in individual organic compounds containing C, H, O, N, and S. This method is more exact than the bomb and VTI methods. There are 6 tables, 5 figures, and 5 references of which are 4 Soviet and 1 English.

Gurevich, I.L. On the Problem of Petroleum Desulfurization 364

The author describes the continuous desulfurization of crudes by means of the MNI adsorption method. Variation of the adsorbent - crude ratio controls the sulfur content of the various fractions. The article gives 3 figures. There are no references.

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OBOLENTSEV, F.D.

"The urgency to intensify researches on the sulphurous petroleum deposits of Bashkiriya"

report presented at the session of the Presidium of the Council for Co-ordination of Scientific Work of the Academies of Sciences of Union Republics and Branches (on Development of Researches on Highly Molecular Compounds) 21 June 1958. (Vest. Ak Nauk SSSR, 1958, No. 9, pp. 101-104)

Chairman of the Bashkiriya Branch of the AS USSR

SEMENOV, N.N., akademik; ARBUZOV, A.Ye., akademik; MAMEDALIYEV, Yu.G.; KARGIN, V.A., akademik; TITOV, N.G., doktor khim.nauk; OBOLENTSEV, R.D., doktor khim.nauk; IMSHENETSKIY, A.A.; SISAKYAN, N.M.

Discussion of the report. Vest. AN SSSR 28 no.8:19-26 Ag '58. (MIRA 11:9)

1. Chlen-korrespondent AN SSSR (for Mamedaliyev, Imshenetskiy, Sisakyan).

(Chemistry, Organic--Synthesis)

AUTHORS: Obolentsev, R. D., Mashkina, A. V. 2o-119-6-38/56

TITLE: The Hydrogenolysis Kinetics of Dibenzothiophene and Octahydrodibenzothiophene Over an Aluminum-Cobalt-Molybdenum Catalyst
(Kinetika gidrogenoliza dibenzotiofena i oktagidrodibenzotiofena nad alyumokobal' tomolibdenovym katalizatorom)

PERIODICAL: Doklady Akademii nauk SSSR, 1958, Vol. 119, Nr 6, pp. 1187-1190 (USSR)

ABSTRACT: The most important modern catalytic working processes of sulfurous mineral oils are based on the hydrogenolysis reactions of organo-sulfuric compounds. They are insufficiently investigated, especially the kinetics mentioned in the title, in spite of the widely spread application of the mentioned catalyst in the mineral oil processing industry. This gap is partly to be closed by the present paper. As the so-called "rest sulfur" mineral-oil products is highly represented by compounds of the thiophene series the authors selected the substances initially mentioned in the title (the second one is called 1, 2, 3, 4, 5, 6,

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The Hydrogenolysis Kinetics of Dibenzothiophene and Octahydrodibenzothiophene Over an Aluminum-Cobalt--Molybdenum Catalyst

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7, 8-octahydro-benzothiophene). Their synthesis is already described. (Refs. 1, 2). As 0,5%-(according to sulfur)-solutions in technical cetane in a flowing-through plant (Ref. 3) with some modifications they were subjected to hydrogenolysis. For the purpose of describing the experimental results (Table 1) the authors tried to employ the kinetic equation by Frost for monomolecular reactions (Ref. 5). Other equations of this type (Refs. 6-9) are mentioned. According to the methodology by S. A. Kazeyev (Ref. 8) the authors found equations which represent the dependence of depth and velocity of both substances mentioned in the title on the duration of contact (Table 2). The correctness of the equations is confirmed by the satisfactory agreement of the experimentally determined and computed depths of the hydrogenolysis (Table 1). Because the clearing up of the dependence of the depth of hydro-desulfonation on the partial hydrogen pressure is important for industry, the authors deduced equations

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of the total dependence of depth and of the velocity of hydrogenolysis of both mentioned substances on the duration of contact and on the mentioned pressure. Because the parameter "b" is practically independent of this pressure, the authors could obtain, after corresponding substitutions the desired equations (8) - (11), which satisfy the experimental results (Table 1). The results of analysis referring to the absorption spectra within the ultraviolet range showed that the total sulfur contained within the liquid catalyst is represented by the not reacted dibenzothiophene and octohydro-dibenzothiophene. Only biphenyl forms the decomposition product of dibenzothiophene, its quantity within the limits of the experimental error agreeing with the quantity of the reacted dibenzothiophene. The hydrogenolysis of the latter proceeds in one stage where biphenyl and hydrogen sulfide form. There are 1 figure, 2 tables, and 10 references, 8 of which are Soviet.

The Hydrogenolysis Kinetics of Dibenzothiophene and Octahydrodibenzothiophene Over an Aluminum-Cobalt--Molybdenum Catalyst 20-119-6-38/56

ASSOCIATION: Bashkirskiy filial Akademii nauk SSSR (Bashkir Branch AS USSR)

PRESENTED: December 11, 1957, by A. V. Topchiyev, Member, Academy of Sciences, USSR

SUBMITTED: December 11, 1957

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OBOLENTSEV, R. D.

11(4) PHASE I BOOK EXPLOITATION SOV/2075

Akademiya nauk SSSR. Bashkirskiy filial, Ufa

Khimiya seraorganicheskikh soyedineniy, soderzhashchikhsya v neftyakh i nefteproduktakh; [materialy III nauchnoy sessii] (Chemistry of Sulphur Organic Compounds Contained in Petroleum and Petroleum Products; [Papers of the Third Scientific Session]) Moscow, Izd-vo AN SSSR, 1959. 376 p. 2,000 copies printed. Errata slip inserted.

Editorial Board: R.D. Obolentsev (Resp. Ed.) Doctor of Chemical Sciences; G.D. Gal'pern, Doctor of Chemical Sciences; Ya. B. Chertkov, Doctor of Technical Sciences; V.V. Panov, Candidate of Technical Sciences; and V.P. Rozhdestvenskiy, Candidate of Chemical Sciences; Ed. of Publishing House: I.I. Brusov; Tech. Ed.: T.P. Polenova.

PURPOSE: This book is intended for chemists, chemical engineers, and technicians specializing in the chemistry of petroleum.

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COVERAGE: The book is a collection of papers presented at the Third Scientific Session on the Chemistry of Organic Sulfur- and Nitrogen Compounds Contained in Petroleum and Petroleum Products. The scientific session was held in Ufa, June 3-8, 1957. The book consists of six sections: 1) Synthesis, characterization, and analysis of organic sulfur compounds; 2) Separation and composition of organic sulfur compounds contained in petroleum and petroleum products; 3) Transformation of organic sulfur compounds by thermal catalysis; 4) Corrosive properties of and tar formation in sulfur-containing petroleum and petroleum products; 5) Uses of organic sulfur compounds and hydrogen sulfide; 6) Physiological properties of organic sulfur compounds. No personalities are mentioned. There are 315 references, of which 179 are Soviet, 118 English, 5 French, 12 German, and 1 Czech.

TABLE OF CONTENTS

Chemistry of Sulphur Organic Compounds (Cont.) SOV/2075

PART I. SYNTHESIS, CHARACTERIZATION, AND ANALYSIS OF ORGANIC SULFUR COMPOUNDS

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PART IV. CORROSIVE ACTIVITY AND TAR FORMATION OF SULFUR-CONTAINING PETROLEUM AND PETROLEUM PRODUCTS

CHIBRIKOVA, Ye.V.; ROZHDESTVENSKAYA, A.A.; OBOLENTSEV, R.D., prof., doktor khim.nauk, glavnyy red.; OLLI, A.I., doktor geol.-miner.nauk, otv.red.; CHEPIKOVA, I.M., red.izd-va; ASTAF'YEVA, G.A., tekhn.red.

[Materials on the paleontology and stratigraphy of Devonian and older deposits of Bashkiria] Materialy po paleontologii i stratigrafii devonskikh i bolee drevnikh otlozhenii Bashkirii. Moskva, Izd-vo Akad.nauk SSSR, 1959. 246 p. (MIRA 13:1)
(Bashkiria--Paleontology, Stratigraphic)

OBOLENTSEV, R. D., GAI PERN, G. D., AIVAZOV, B. V., BERINGER, N. N. KARAULOVA, E. N., LUKYANITSA, V. G., RATOVSKAYA, A. A., TIMOFEYEV, V. D. (SECTION V)

"Composition of Sulfur- and Nitrogen-Organic Compounds Contained in the Oil of the Eastern Areas in the Soviet Union."

Report submitted at the Fifth World Petroleum Congress, 30 May - 5 June 1959. New York.

OBOLENTSEV, R.D., doktor khim. nauk, otv. red.; MASHKINA, A.V., kand. khim. nauk, red.; NOZHKINA, I.A., kand. khim. nauk, red.; ROZHDESTVENSKIY, V.P., kand. khim. nauk, red.; GLADKOVA, L.K., red.; SIDOROV, V.V., red.; SHAFIN, I.G., tekhn. red.

[Chemistry of sulfur and nitrogen organic compounds in petroleum and petroleum products] Khimiia sera- i azotoorganicheskikh soedinenii, soderzhashchikhsia v neftiakh i nefteproduktakh. Ufa. Vol.3. 1960. 337 p. (MIRA 14:5)

1. Akademiya nauk SSSR. Bashkirskiy filial, Ufa. Otdel khimii. 2. Bashkirskiy filial AN SSSR, Otdel khimii (for Obolentsev)
(Petroleum--Analysis) (Sulfur organic compounds)
(Nitrogen organic compounds)

OBOLENTSEV, R.D.

PHASE I BOOK EXPLOITATION SOV/4941

Mezhvuzovskoye soveshchaniye po khimii nefti, Moscow, 1956.

Sbornik trudov Mezhvuzovskogo soveshchaniya po khimii nefti (Collection of Transactions of the Inter-University Conference on Petroleum Chemistry) [Moscow] Izd-vo Mosk. univ., 1960. 313 p. Errata slip inserted. 1,600 copies printed.

Organizing Committee of the Conference: Chairman: B. A. Kazanskiy, Academician; Vice-Chairman: S. I. Khromov, Docent; O. M. Panchenkov, Professor; A. F. Plate, Professor; Secretary: Ye. S. Balenkova; Scientific Worker. Editorial Board: Resp. Ed.: A. F. Plate; I. V. Gostunskaya, I. N. Tits-Skvortsova, L. A. Erivanskaya.

PURPOSE: This collection of articles is intended for the teaching staff of universities and schools of higher education, training specialists for the petroleum and petroleum-refining industries.

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COVERAGE: The collection includes articles dealing with the present state of the petroleum industry, the scientific research problems in petroleum chemistry, the chemistry of petroleum, the composition of petroleum and petroleum products, the scientific principles of refining petroleum into motor fuels and lubricants, and the manufacture of synthetic products from hydrocarbon gases and petroleum. One article discusses the effect of chemical composition and additives on fuel combustion in jet engines. The material was presented at the Inter-University Conference on Petroleum Chemistry, held at the Moscow State University imeni M. V. Lomonosov November 26-28, 1956. No personalities are mentioned. References accompany most of the articles.

TABLE OF CONTENTS: None given

The authors and the titles of articles are as follows:

Introduction by B. A. Kazanskiy, Academician

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S/081/62/000/005/038/112
B151/B101

AUTHORS: Obolentsev, R. D., Bukharov, V. G., Pozdnyakova, T. Ye., ~~Nialykina~~, L. A., Bukalo, L. A., Pototskaya, A. Ye.

TITLE: The synthesis of mono-substituted thiophanes

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 5, 1962, 263-264, abstract 5Zh230 (Sb. "Khimiya sera- i azotorgan. soyedineniy, soderzhashchikhsya v neftyakh i nefteproduktakh". v. 3. Ufa, 1960, 9-17)

TEXT: A general method is put forward for the synthesis of α-substituted thiophanes, starting from alkylfurylcarbinols, according to the following scheme;

$RCH(OH)\dot{C}=CHCH=\overline{CHO}$ (I)→$RCO-CH_2CH_2COOC_2H_5$ (II)→$RCH(OH)CH_2CH_2CH_2OH$ (III)→

→$RCHBrCH_2CH_2CH_2Br$ (IV)→$R\dot{C}HCH_2CH_2CH_2S$ (V). I is converted by heating for 2.5 - 3 hrs. in abs. C_2H_5OH containing 0.3 - 0.5% HCl gas (in the

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The synthesis of mono-substituted ...

case of high mol. wt. R the heating is carried out for 0.5 hrs, 4-9% HCl gas) with yields of 35 - 60%, into etnyl esters II (IIa-f) (here and later are given the substance, R, b. p. in °C/mmHg, $n^{20}D$, d_4^{20}): IIa, $CH_3C(CH_3)_2CH_2$, 89-91/4, 1.4346, 0.9293; b, $CH_3CH_2C(CH_3)_2CH_2$, 104-105/4, 1.4410, 0.9562; c, $CH_3(CH_2)_5$, 113-115/2, 1.4370, 0.9440; d, $CH_3(CH_2)_7$, 131-132/2, 1.4403, 0.9317; e, $CH_3(CH_2)_8$, 145-146/3, 1.4430, 0.9256; f, CH_3-$(CH_2)_{10}$, -, m. p. 25-27°C, -, -. The II obtained are reduced with a two-fold excess of $LiAlH_4$ to the corresponding III (IIIa-i): IIIa, $CH_3C(CH_3)_2CH_2$, 112-114/3, 1.4545, 0.9319; b, $CH_3CH_2C(CH_3)_2CH_2$, 123-124/3, 1.4637, 0.9373; c, $CH_3(CH_2)_5$, 139-140/3.5, 1.4558, 0.9249; d, $CH_3(CH_2)_7$,-, m. p. 46-46.5°C, -, -; e, $CH_3(CH_2)_8$,-, m. p. 41.5-42°C, -, -; f, $CH_3(CH_2)_{10}$,-, m. p. 59-60°C, -, -; g, 2-$C_{10}H_7$ -, m. p. 88-89°C, -, -; h, 4-diphenyl, -, m. p. 80°C, -, -; i, cyclo-$C_5H_{11}CH_2$, -. m. p. 59.5-60.0°C,

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The synthesis of mono-substituted ...

-, -. The III glycols are dissolved in glacial CH_3COOH and the solution saturated with dry HBr at 100-120°C and then fractionated, when the IV (IVa-i) are obtained; IVa, $CH_3C(CH_3)_2CH_2$, 12?-126/15, 1.4665, 1.3646; b, $CH_3CH_2C-(CH_3)_2CH_2$, 92-102/2, 1.4962, 1.3623; c, $CH_3(CH_2)_5$, 122-123/3, 1.4940, 1.3607; d, $CH_3(CH_2)_7$, 137-139/2, 1.4502, 1.2976; e, $CH_3(CH_2)_8$, 157-159/2.5, 1.4865, 1.2633; f, $CH_3(CH_2)_{10}$, 180-182/3, 1.4803, 1.2201; g, 2-$C_{10}H_7$, -, m. p. 54-56°C, -, -; h, 4'-diphenyl, -, m. p. 84-85°C, -, -; i, cyclo-$C_6H_{11}CH_2$, 132-133/1.5, 1.5202, 1.4310. On boiling the dibromides IV for 3 hrs with a 50% water-alcohol solution of Na_2S there are formed, with yields of 80-90%, the V (Va-k): Va, $CH_3C(CH_3)_2CH_2$, 202-203/760, 1.4812, 0.9155; b, $CH_3CH_2C(CH_3)_2CH_2$, 107-108/17, 1.4862, 0.9272; c, $CH_3(CH_2)_5$, 240-241/760, 1.4823, 0.9095, d, $CH_3(CH_2)_7$, 275.5 - 276/760, 1.4793, 0.8992; e, $CH_3(CH_2)_8$, 292-293/760, 1.4792, 0.8940;

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The synthesis of mono-substituted ...

f, $CH_3(CH_2)_{10}$, 326.5 - 327/760, 1.4786, 0.8936, g, 2-$C_{10}H_7$, m. p. 74-75°C, -, -, -; h, 4'-diphenyl, m. p. 59-60° , -, -, -; i, cyclo-C_6H_{11}-CH_2, 86-87/2, 1.5135, 0.9811; k, C_6H_5-CH_2, 109-110/2, 1.5710, 1.0577. With the method given it was not possible to obtain V_k since the original phenylfurfurylcarbinol on boiling with an alcohol solution of HCl resinifies and the corresponding dibromide was obtained in another way. (R. Paul, Compt. rend., 1936, 202, 1444). The glycols IIIg and IIIh were obtained by the reductions of the corresponding β-(2-naphthoyl) and β-(4-biphenyloyl)-propionic acids, synthesized by the condensation of the corresponding hydrocarbons with the succinic anhydride using the Friedel-Crafts reaction. The 3-alkylthiophanes were obtained by another method:
$H_5C_2COCH_2CH(COOC_2H_5)_2$ (VI)⟶$H_5C_2COCH_2CR(COOC_2H_5)_2$ (VII)⟶
⟶$RCH(COOC_2H_5)CH_2COOC_2H_5$ (VIII)⟶$RCH(CH_2OH)CH_2CH_2CH_2OH$ (IX)⟶
⟶$RCH(CH_2Br)CH_2CH_2Br$ (X)⟶$RCHCH_2CH_2SCH_2$ (XI). The Na derivatives of VI are condensed in the usual way with halogen alkyls and yields of 80-90% of VII are obtained. These are saponified, decarboxylated and esterified

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The synthesis of mono-substituted ...

when VIII (VIIIa-d) are obtained in a yield of 70-90%. VIIIa, $(CH_3)_2CHCH_2$, 96-98/2, 1.4260, 0.9710; b, CH_3CH_2-$CH(CH_3)CH_2$, 101-103/2, 1.4300, 0.9633; c, $CH_3(CH_2)_4$, 96-97/1.5, 1.4510, 0.9625; d, $CH_3(CH_2)_7$, 130-131/1, 1.4305, 0.9455. VIII is reduced with $LiAlH_4$ (1.25 moles) and (IXa-d) are distilled off: IXa, $(CH_3)_2CHCH_2$, 118-120/1.5, 1.4525, 0.9396; b, $CH_3CH_2CH(CH_3)CH_2$, 129-130/2.5, 1.4550, 0.9289; c, $CH_3(CH_2)_4$, 132-134/3, 1.4560, 0.9299; d, $CH_3(CH_2)_7$, 161-162/2, 1.4590, 0.9157. From the IX obtained by the method described above the X (Xa-d) are obtained: Xa, $(CH_3)_2CHCH_2$, 75-76/1.5, 1.4983, 1.4751; b, $CH_3CH_2CH(CH_3)CH_2$, 102-103/2.5, 1.4975, 1.4205; c, $CH_3(CH_2)_4$, 114-116/3, 1.4975, 1.4144; d, $CH_3(CH_2)_7$, 126-129/1, 1.4910, 1.3078. The X are converted in the usual way into XI (XIa-d); XIa $(CH_3)_2CHCH_2$, 200-201, 1.4850, 0.9216; b, $CH_3CH_2CH(CH_3)CH_2$, 221.5-222, 1.4824, 0.9168; c, $CH_3(CH_2)_4$, 229.5-230, 1.4842, 0.9164; d, $CH_3(CH_2)_7$, 282.5-283, 1.4808, 0.9057. The yields in XI were 84-93%

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B151/B101

The synthesis of mono-substituted ...

based on X and 30-40% based on VI. Abstracter's note: Complete translation.

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OBOLENTSEV, R.D.; BUKHAROV, V.G.; GERASIMOV, M.M.

Sulfoxides and sulfones of some monosubstituted thiophanes. Khim. sera i azotorg.soed. sod.v neft.i nefteprod. 3:35-41. '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Sulfoxides) (Sulfone) (Thiphene)

OBOLENTSEV, R.D.; BUKHAROV, V.G.; GERASIMOV, M.H.

Reduction of thiophane sulfones and its homologs by means of aluminum lithium hydrides. Khim.sera-i azotorg.soed.sod.v neft.i nefteprod. 3:43-49 '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Sulfone) (Aluminum lithium hydride) (Reduction, Chemical)

OBOLENTSEV, R.D.; BUKHAROV, V.G.; FAYZULLINA, N.K.

Complex compounds of some cyclic and aliphatic sulfides with mercury chloride. Khim.sera-i azotorg.soed.sod.v neft.i nefteprod. 3:51-65 '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Sulfur organic compounds) (Mercury chloride)

OBOLENTSEV, R.D.; BUKHAROV, V.G.; FAYZULLINA, N.K.

Iodomethylates of some cyclic and aliphatic sulfides. Khim.sera-i azotorg.soed. sod.v neft.i nefteprod. 3:67-73. '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Sulfide) (Iodomethylation)

31545
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B102/B108

5 5620 2220

AUTHORS: Zimina, K. I., Obolentsev, R. D., Polyakova, A. A., Khmel'nitskiy, R. A.

TITLE: Mass spectra of some homologs of thiophane

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 22, 1961, 12-13, abstract 22B72 (Sb. "Khimiya sera-i azotorgan. soyedineniy, soderzhashchikhsya v neftyakh i nefteproduktakh", Ufa, v. 3, 1960, 81-92

TEXT. The mass spectra of α-alkyl thiophanes with radicals of normal structure from C_1 to C_6 were studied by means of an MC-1 (MS-1) mass spectrometer. Total ionization caused by 70-ev electrons was studied as dependent on the molecular weight of the substance investigated: It is shown that the total quantity of molecules and fragmentary ions increases linearly with increasing molecular weight. The total ionization value measured for thiophenes makes it possible to carry out an analysis of the structural groups of heterocyclic compounds. The ionization potentials of thiophanes were determined approximately. They were found to decrease

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(from 9.5 ev for C_1 to 8 ev for C_6) with increasing length of the chain of the alkyl radical. The mass spectra of the alkyl thiophanes were all characterized by the presence of an intense peak at the mass 87 which permits identifying these compounds. [Abstracter's note: Complete translation.]

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OBOLENTSEV, R.D.; LYUBOPYTOVA, N.S.; MAKOVA, Ye.A.

Absorption spectra in the ultraviolet of some cyclic sulfides, thianthrene, and 2-ethylthiophene. Khim.sera.i azotorg.soed.sod.v neft.i nefteprod. 3:93-104 '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Sulfide—Spectra) (Thianthrene—Spectra) (Thiophene—Spectra)

OBOLENTSEV, R.D.; KOTOV, Yu.I.; CHELOV, Ye.N.

Vibrational spectra of sulfides. Khim.sera-i azotorg.soed.sod.v neft. nefteprod. 3:105-114 '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Sulfide—Spectra)

OBOLENTSEV, R.D.; RATOVSKAYA, A.A.; TIMOFEYEV, V.D.

Sulfide sulfur in some crude oils of Bashkiria. Khim.sera i azotorg. soed.sod.v neft.i nefteprod. 3:167-172 '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Bashkiria—Petroleum—Analysis) (Sulfur—Analysis)
(Sulfide)

OBOLENTSEV, R.D.; AYVAZOV, B.V. ; TITOVA, K.V.

Comparative characteristics of various silica gel samples based on their relations to sulfur organic compounds contained in some fuels. Khim.sera-i azotorg.soed.sod.v neft.i nefteprod. 3:211-217 '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Silica) (Adsorption) (Sulfur organic compounds)

OBOLENTSEV, R.D.; AYVAZOV, B.V.; GALEYEVA, G.V.; CHELOV, Ye.N.

Composition of sulfur organic compounds in a straight-run fuel produced from Tuymazy and Bavly oils. Khim.sera-i azotorg.soed.sod.v neft.i nefteprod. 3:241-250 '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Sulfur organic compounds) (Fuel--Analysis)

OBOLENTSEV, R.D.; AYVAZOV, B.V.; TITOVA, K.V.

Role of elementary sulfur in the formation of hydrogen sulfide during the heating of curde oils. Khim.sera-i azotorg.soed. sod.v neft. i nefteprod. 3:253-259 '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Petroleum—Thermal properties) (Hydrogen sulfide)
(Sulfur)

OBOLENTSEV, R.D.; GABDULLINA, L.N.

Kinetics of transformation of 2, 8-dimethyl-5-thianonane and 5, 7-dipropyl-6-thiaundecane in the presence of an aluminosilicate catalyst. Khim.sera-i azotorg.soed.sod.v neft¸i nefteprod. 3:261-270 '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Nonane) (Undecane)
(Sulfur organic compounds)

S/081/62/000/005/057/112
B151/B101

AUTHORS: Obolentsev, R. D., Dronov, V. I.

TITLE: The kinetics of transformation of some monocyclic sulfides and 2-etylthiophene in the presence of a globular aluminosilicate catalyst

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 5, 1962, 206, abstract 5Zh56 (Sb. "Khimiya sera- i azotorgan. soyedineniy, soderzhashchikhsya v neftyakh i nefteproduktakh" v. 3 Ufa, 1960, 271-294)

TEXT: The kinetics of the transformation of cis- and trans-2,5- dimethyl thiophanes (I), 2-etnylthiophane (II), 2-methyl thia-cyclohexane (III) thiacycloheptane (IV), trans-2,5-di-n-propylthiophane (V) and 2-ethylthiophene (VI) in the presence of a globular aluminosilicate catalyst in a flow system, at atmospheric pressure, is studied. For I - IV the reaction is carried out at 350 - 450°C and a volume velocity (VV) from 1 to 40 hrs^{-1}, for VI at 450-550°C and VV 0.3 - 4 hrs^{-1}, at a

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concentration of 0.45% on the sulfur in a C_6H_6 medium. The experiments with V are carried out in cetane, decalin and α-methyl-naphthalene at 500 - 400°C, at VV from 2.5 to 80 hrs^{-1}. The main sulfur-containing product of all the catalytic transformations is H_2S. The kinetic equations are derived. The dependence of the parameters of these equations on the reciprocal of the absolute temperature is found. The mean velocity of transformation of the monocyclic sulfides depends on their structure and increases with increasing molecular weight. The rate of transformation of 2- alkylthiophanes is lower than that of the isomeric 2,5-dialkylthiophanes. For the isomeric monocyclic sulfides with 5 and 6 membered rings the rates are almost equal, and higher than that for 7 membered rings. With the cis- and trans-isomers of 2,5-dialkylthiophanes the rates are very close together. The difference in the rates of transformation of the monocyclic sulfides is reflected in the composition of the monocyclic sulfides contained in the distilled products from catalytic cracking which enrich in low mol. wt. sulfides and 2-alkylthiophanes at the expense of high mol. wt. sulfides and 2,5-dialkylthiophanes. The desulfuration of petroleum products in the catalytic cracking process depends on their hydrocarbon composition, which

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The kinetics of transformation of ...

is borne out by the different rates of transformation of V depending on the solvent. The aluminosilicate catalyst can be used for getting rid of monocyclic sulfides from their mixtures with thiophenes, the rate of transformation of which is considerably lower, as shown in the case of VI.
[Abstracter's note: Complete translation.]

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OBOLENTSEV, R.D.; MASHKINA, A.V.

Hydrogenolysis of sulfur organic compounds under conditions of hydrofining. Khim.sera-i azotorg.soed.sod.v neft.i nefteprod 3:295-329 '60. (MIRA 14:6)

1. Bashkirskiy filial AN SSSR, Otdel khimii.
(Sulfur organic compounds) (Petroleum—Refining)

S/081/61/000/021/014/094
B102/B138

AUTHORS: Obolentsev, R. D., Torikov, D. M.

TITLE: The kinetics of the hydrogenolysis of compounds of the thiophene and thiophane series with a platinum catalyst

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 21, 1961, 52, abstract 21B420 (Sb. "Khimiya sera- i azotorgan. soyedineniy, soderzhashchikhsya v neftyakh i neftoproduktakh", Ufa, v. 3, 1960, 333 - 343)

TEXT: The reaction kinetics of the hydrogenolysis of thiophene, 2-ethylthiophene, 2-butylthiophene, 2,5 -diethylthiophene, 2,5 -dibutylthiophene, 2-butylthiophane and cis-2,5 -dimethylthiophane were studied, in the presence of a platforming catalyst at 340, 400 and 500°C, a total pressure of 400 atm, a molar hydrogen to raw material ratio of 5:1 and a contact time of 0.5 - 8 sec. Kinetic equations are proposed which describe the dependence of depth and rate of hydrogenolysis on contact time, and relations are derived for the temperature dependence of the parameters of

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B102/B138

the kinetic equations. The reaction energies of hydrogenolysis and the kinetic coefficients of the activity of the reactions are determined. [Abstracter's note: Complete translation.]

Card 2/2

CHERTKOV, Ya.B.; ZRELOV, V.N.; OBOLENTSEV, R.D.

Thermal stability of sulfur compounds and their effect on the performance characteristics of fuels. Khim.sera-i azotorg.soed.v neft.i nefteprod. 3:461-468 '60. (MIRA 14:6)

1. Nauchno-issledovatel'skiy institut goryuche-smazochnykh materialov, Bashkirskiy filial AN SSSR.
(Sulfur organic compounds—Thermal properties)
(Fuel—Testing)

YENIKEYEVA, M.Z.(Ufa); KRIVOLAPOV, S.S.(Ufa); ~~SOLENTSEV~~, R.D.(Ufa); ROZHDESTVENSKIY, V.P. (Ufa)

Reduction roasting of rich iron ores by mixtures of petroleum gas and water vapor. Izv. AN SSSR. Otd.tekh.nauk. Met.i topl. no.5:19-24 S-O '60. (MIRA 13:11)
(Iron ores) (Ore dressing)

S/079/60/030/04/47/080
B001/B002

AUTHORS: Zimina, K. I., Polykova, A. A., Khmel'nitskiy, R. A., Obolentsev, R. D.

TITLE: Mass-spectrometric Investigation of Some Homologs of Thiophane

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 4, pp. 1264-1268

TEXT: Only a small number of reports on the mass spectra of sulfur compounds had been hitherto published. Detailed investigations were only carried out with respect to a series of thiophenes, whose spectroscopic data were, as expected, similar to those of alkyl benzenes (Ref. 2). In the present paper the results of mass-spectrometric investigations of homologous α-alkylthiophanes exhibiting radicals of normal structure (C_1 - C_6) were described. The spectrometric investigation by means of the already earlier (Ref. 3) modified mass spectrometer MC-1 (MS-1) is described in detail. The distribution of the mass intensities in the spectra, the values of the relative sensitivity, and the dependence of these values on

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Mass-spectrometric Investigation of Some Homologs of Thiophane

S/079/60/030/04/47/080
B001/B002

the molecular weight were determined. The complete ionization was computed. It was shown that an identification of the structures, and a qualitative analysis of α-alkylthiophane mixtures is possible. The accuracy of the quantitative analysis of the mixtures is relatively 10 - 15%. The data given, are partly provided by the Vsesoyuznyy nauchno-issledovatel'skiy institut neftyanoy promyshlennosti (All-Union Scientific Research Institute of Petroleum Industry). There are 3 figures, 2 tables, and 5 references, 1 of which are Soviet.

SUBMITTED: March 28, 1959

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5.3200
~~5(3)~~ 5.3300(B)

AUTHORS: Obolentsev, R. D., Dronov, V. I. SOV/20-130-1-27/69

TITLE: Transformation Kinetics of Some Monocyclic Sulfides on an Aluminosilicate Catalyst

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol 130, Nr 1, pp 98-101 (USSR)

ABSTRACT: The problem mentioned in the title is of importance in connection with the catalytic cracking of petroleum containing much sulfur. With their paper, the authors wanted to close a gap in the respective publications. For this purpose, they used cis- and transisomers of 2,5-dimethylthiophane and 2,5-di-n-propylthiophane, 2-ethylthiophane, 2-n-hexylthiophane, 2-methylthiacyclohexane, thiacycloheptane, and 2-ethylthiophene. A continuously working laboratory apparatus was used for the experiments which were carried out at atmospheric pressure. The volume velocity was between 1 and 80. The sulfides were catalyzed in benzene and some other solvents as 0.45% solutions (computed with respect to sulfur). The catalyst used was an aluminosilicate catalyst with the activity index 33. A sulfur balance was made for each experiment. The authors proved that H_2S is the most

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important sulfur-containing product of the transformations mentioned. The quantity of mercaptane sulfur in the catalyzates does not exceed 2.5% computed with respect to the sulfur content in the initial raw material. The sulfur content in the coke does not exceed 3% of the sulfur in the initial solution. Equations (1) and (2) suggested in a general form by A. V. Frost (Refs 1, 4) and S. A. Kazeyev (Refs 2, 5) were used for the kinetic characteristics of the above transformations. Table 1 shows the characteristic values determined for the kinetic equations of the above substances. Their degrees of transformation were computed in dependence on the contact time at 350, 400 and 450° C. Figure 1 shows such a dependence at 450° as an example. It shows that the points determined experimentally lie rather exactly on the curves computed. In a short contact time, differences in the average transformation rates of the cyclic sulfides occur. This rate increases with the molecular weight of the sulfide. From this and other differences found, the authors conclude that this phenomenon certainly influences the composition of the cyclic sulfides contained in the distillation products of catalytic cracking. These products are enriched by low-molecular sulfides and 2-alkylthiophanes at the expense of the content in

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high-molecular sulfides and 2,5-dialkylthiophanes. Thus, it was shown that an aluminosilicate catalyst can be used for eliminating monocyclic sulfides from their mixture with thiophenes. The factors of diffusion inhibition computed according to A. Ya. Rozovskiy and V. V. Shchekin (Ref 6) showed that - depending on the grain size of the catalyst - cis-2,5-dimethylthiophane is either transformed in the inner diffusion range or in the transition range. The authors assume that distillates with the lowest sulfur content are produced in cracking in a suspension with a dustlike catalyst. Figure 2 shows that trans-2,5-di-n-propylthiophane, dissolved in benzene and α-methyl naphthalene, is most rapidly transformed. 2,5-di-n-propylthiophane in cetane is the slowest to transform. A higher content of isooctylene in the isooctane-isooctylene mixture reduces the transformation intensity of cis-2,5-dimethylthiophane (Fig 3). The degree of desulfurization can be

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determined from the kinetic characteristics of the sulfide mixtures in petroleum products. The total intensity of transformation of a sulfide mixture follows the rule of additivity (Fig 4). There are 4 figures, 1 table, and 6 Soviet references.

ASSOCIATION: Bashkirskiy filial Akademii nauk SSSR (Bashkiriya Branch of the Academy of Sciences, USSR)

PRESENTED: July 7, 1959, by A. V. Topchiyev, Academician

SUBMITTED: July 6, 1959

80001

5.3620

AUTHORS: Obolentsev, R. D., Mashkina, A. V.

S/020/60/131/05/030/069
B011/B117

TITLE: Kinetics of the Reactions of Hydrodesulfuration

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol 131, Nr 5, pp 1092-1095 (USSR)

TEXT: In their paper, the authors report on the results of the systematic study of the kinetics of hydrogenolysis for 14 sulfides and thiophenes in the presence of an aluminum-cobalt-molybdenum catalyst (Al-Co-Mo). Experimental methods used were described in reference 4. Figure 1 shows, as an example, the curves of the dependence of the conversion degree on the time of contact for 2,4,6,8-tetramethyl-5-thiononane. The shape of these curves is characteristic of all compounds investigated here. Hydrogenolysis is well-defined by equations suggested in a general form by Frost ($v_o \ln \frac{1}{1-y} = \alpha+\beta v_o y$) and Kazeyev ($\ln \frac{D}{D-M} = a\tau^b$) ($v_o$ being the average feeding rate of the compound used to the reaction vessel per 1 g of the catalyst per 1 hour; y the intensity of hydrogenolysis in fractions of unity; α and β parameters; τ time of contact in seconds, M the intensity of hydrogenolysis in %; D the limit of M for $\tau \to \infty$, a and b parameters). The parameters of these equations for 14 compounds investigated are given in table 1. From an analysis of these parameters, it follows that the organic compounds of

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sulfur form as to their rate of hydrogenolysis, the following sequence with increasing activity holds: A, B, V, G, D, Ye, Zh, Z, K, L, M, N, O, P. The values of the parameter α are proportional to the rate constants of the hydrogenolysis reaction of these compounds at 375°. They are related to each other in the following way: (A, B, V, G, D) : (Ye, Zh) : (Z, K) : (L, M, N, O) : P = = 1 : 2 : 3 : 4 : 7. The possibility of obtaining a selective hydrodesulfuration of petroleum products on the Al-Co-Mo catalyst is based on this dependence. In addition, this catalyst may be used to perform group analyses of organic compounds of sulfur. This dependence makes it possible, moreover, to predict the type of products formed by hydrogenolysis. The hydrogenolysis rate of organic compounds of sulfur obeys the law of additivity (curves in Fig 2). Thereby, the prediction of the composition of organic compounds of sulfur formed when petroleum products are hydrodesulfurized is made possible. The authors intended to study the influence of the reaction products on the rate of hydrogenolysis. Dibenzo thiophene solved in cetane with diphenyl and H_2S added was therefore subjected to hydrolysis. Maximum intensity and rate of hydrogenolysis are rapidly reduced by the addition of biphenyl to the initial solution of dibenzo thiophene, but are practically independent of the H_2S added (Fig 3). Dibenzo thiophene together with its derivatives represents the major part of the so-called "residual sulfur".

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Results obtained are of interest in the processing of petroleum. From table 2, it is obvious that hydrodesulfuration should be performed in the dispersed layer for the best results. Moreover, data obtained by the authors can contribute to the development of new ways of obtaining hydrodesulfurizing catalysts. The main products of hydrogenolysis are H_2S and the corresponding hydrocarbon. Monocyclic hydrocarbons are not hydrogenated practically under the conditions given. The Al-Co-Mo catalyst can be used to identify organic compounds of sulfur by means of their hydrolysis products (analogously to Raney nickel). There are 3 figures, 2 tables, and 4 references, 2 of which are Soviet.

ASSOCIATION: Otdel khimii Bashkirskogo filiala Akademii nauk SSSR (Department of Chemistry of the Bashkiriya Branch of the Academy of Sciences, USSR)

PRESENTED: December 8, 1959, by A. V. Topchiyev, Academician

SUBMITTED: December 8, 1959

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OBOLENTSEV, R.D., prof., doktor khim. nauk, otv. red.; GLADKOVA, L.K., red.; DRONOV, V.I., red.; KALANTAR, N.G., kand. tekhn. nauk, red.; MIKHEYEV, G.M., red.; POZDEYEV, N.M., kand. fiz.-mat. nauk, red.; KLEYMENOVA, K.F., vedushchiy red.; FEDOTOVA, I.G., tekhn. red.

[Materials of the Scientific Session on Chemistry of Sulfur- and Nitrogen Organic Compounds Contained in Petroleum and Petroleum Products] Materialy Nauchnoy sessii po khimii sera- i azotorganicheskikh soedineniĭ, soderzhashchikhsia v neftiakh i nefteproduktakh. 5th, Ufa, 1959. Moskva, Gos. nauchno-tekhn. izd-vo neft. i gorno-toplivnoi lit-ry. Vol.4. [Chemistry ofsulfur organic compounds contained in petroleum and petroleum products] Khimiia seraorganicheskikh soedinenii, soderzhashchikhsia v neftiakh i nefteproduktakh. 1961. 278 p. (MIRA 14:9)

1. Nauchnaya sessiya po khimii sera- i azotorganicheskikh soyedineniy, soderzhashchikhsia v neftiakh i nefteproduktakh. 5th, Ufa, 1959.
2. Bashkirskiy filial AN SSSR, otdel khimii (for Obolentsev).
(Petroleum—Analysis) (Sulfur organic compounds)

PHASE I BOOK EXPLOITATION SOV/5769

Obolentsev, Roman Dmitriyevich, and Anna Vasil'yevna Mashkina

Gidrogenoliz seraorganicheskikh soyedineniy nefti (Hydrogenolysis of Organic Sulfur Petroleum Compounds) Moscow, Gostoptekhizdat, 1961. 143 p. 2,100 copies printed.

Executive Ed.: O. M. Yenisherlova; Tech. Ed.: A. S. Polosina.

PURPOSE: This book is intended for scientific workers and engineers at research institutes, design and planning organizations, and petroleum refineries, and can also be used by students in advanced courses in schools of higher technical education specializing in petroleum engineering.

COVERAGE: The book systematizes and describes reactions of the hydrogenolysis of organic sulfur compounds of the type present in petroleum crudes. Information is also given on reaction thermodynamics, kinetics, and the mechanism of reactions which constitute the theoretical basis of the hydrorefining process. A significant part of the data can serve as reference material for designing, planning, and operating hydrorefining installations at petroleum-refining plants

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No personalities are mentioned. There are 116 references: 67 Soviet, 46 English, and 3 German.

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S/081/62/000/005/039/112
B151/B101

AUTHORS: Obolentsev, R. D., Bukharov, V. G., Baisheva, A. U.

TITLE: Synthesis of cis- 2,5-dibenzylthiophane

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 5, 1962, 264, abstract 5Zh237. (Sb. "Khimiya seraorgan. soyedineniy, soderzhashchikhsya v neftyakh i nefteproduktakh. v. 4." M., Gostoptekhizdat, 1961, 20-23)

TEXT: A new method is developed for the synthesis of the stereoisomers of 2,5-disubstituted thiophane (I thiophane). By bromination of adipic acid (II) a mixture (90%) of almost equal amounts of the isomers of 2.5-dibromo-II is obtained, and these are separated by the ethyl esters (EE) into the crystalline meso- and the liquid racemic forms in a yield of 30%. The latter are cyclized by the action of $(NH_4)_2S$ in a mixture of water and alcohol and acetone in the cold to form the isomeric EE of thiophane-2,5-dicarboxylic acids (III) in a yield of 64-65%. On reduction of the trans- and cis-III the corresponding glycols are easily

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obtained (yield - 100%). These are unstable to heat. The halogenides prepared from them (PBr_3 or $SOCl_2$ in the cold) are of low reactivity. For instance $LiAlH_4$ reduces them only with difficulty to 2,5-dimethyl-I in a yield of 35%. Li-organic compounds react more energetically. With C_6H_5Li, from cis-2,5-dibromo-methyl-1 firstly cis-2,5-dibenzyl-I is obtained in a yield of 55%. Another possible method: III + CH_3MgI (or C_2H_5MgBr) → 2.5-bis-dialkyl-oxymethyl-I (yield 42-75%) → 2.5 bis-dialkyl chloro-methyl-I (IV) HCl (acid) yield 75% did not give results, since IV does not form an Mg-organic compound and is only with difficulty reduced with a large excess of $LiAlH_4$ even with prolonged boiling. The substances so formed, yield 50%, probably consist of a mixture of the corresponding 2,5-bis-dialkylmethyl-I and the products of dehydrochlorination of IV. Abstracter's note: Complete translation.

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S/081/62/000/006/069/117
B149/B108

AUTHORS: Obolentsev, R. D., Timofeyev, V. D., Ratovskaya, A. A., Baykova, A. Ya., Rafikova, L. G., Gavrilova, L. D.

TITLE: Group-composition of organic sulfur compounds in petroleum from the Bashkirskaya ASSR

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 6, 1962, 527, abstract 6M135 (Sb. "Khimiya seraorgan. soyedineniy, soderzhashchikh-ya. v neftyakh i nefteproduktakh. v. 4", M., Gostoptekhizdat., 1961, 103 - 112)

TEXT: The total sulfur, sulfide and elemental sulfur content of crude petroleum from various deposits were determined, the former by double combustion, the two latter by anode polarography with solid electrodes. In addition, the distribution of organic sulfur compounds according to fractions with onset of boiling at 120, 120 - 200, 200 - 250, and 250-300°C from a series of petroleums was studied. The sulfide sulfur in the fractions was determined by the iodine complex method, the mercaptan sulfur by the Grimms method. Elemental sulfur was found in only one of

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