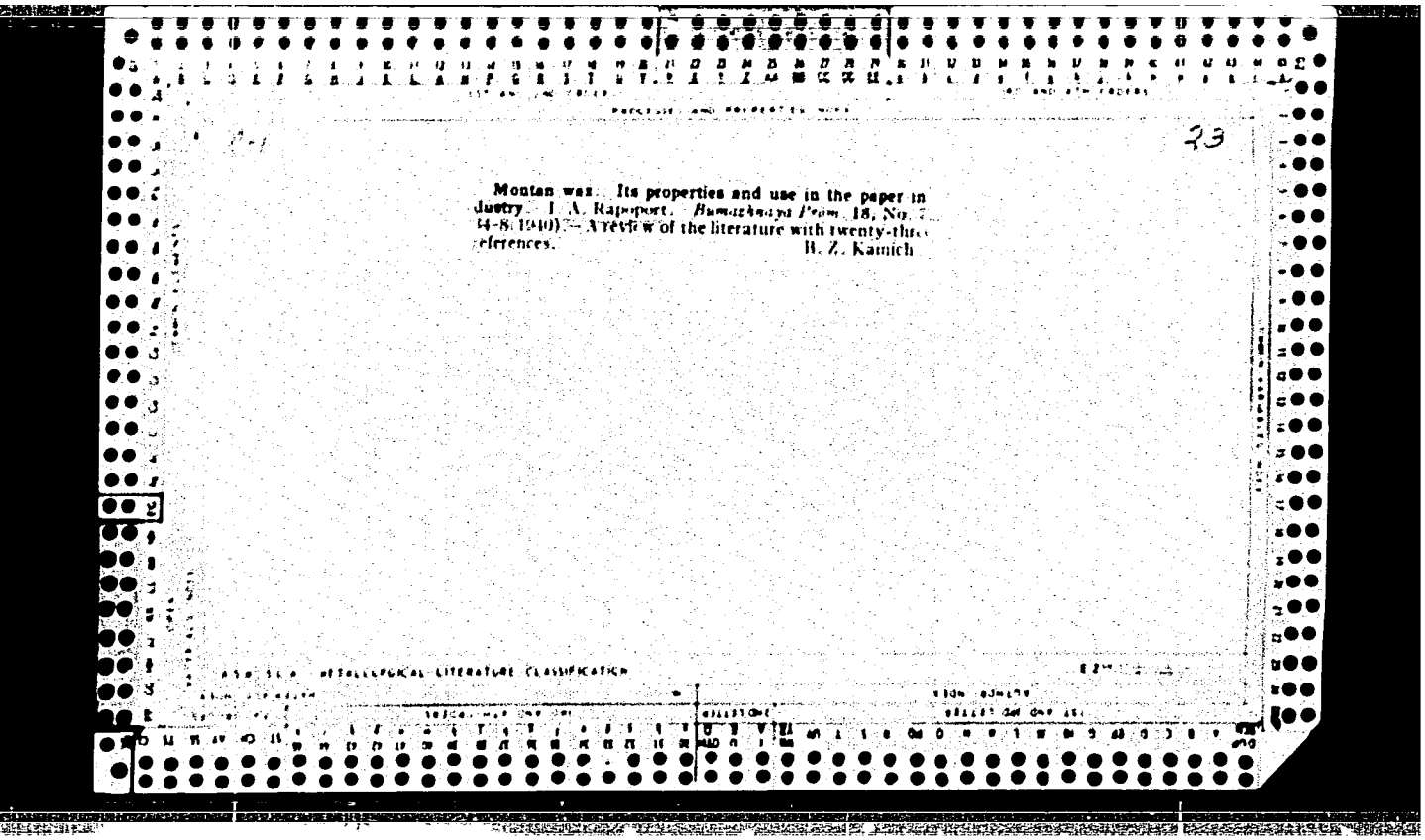
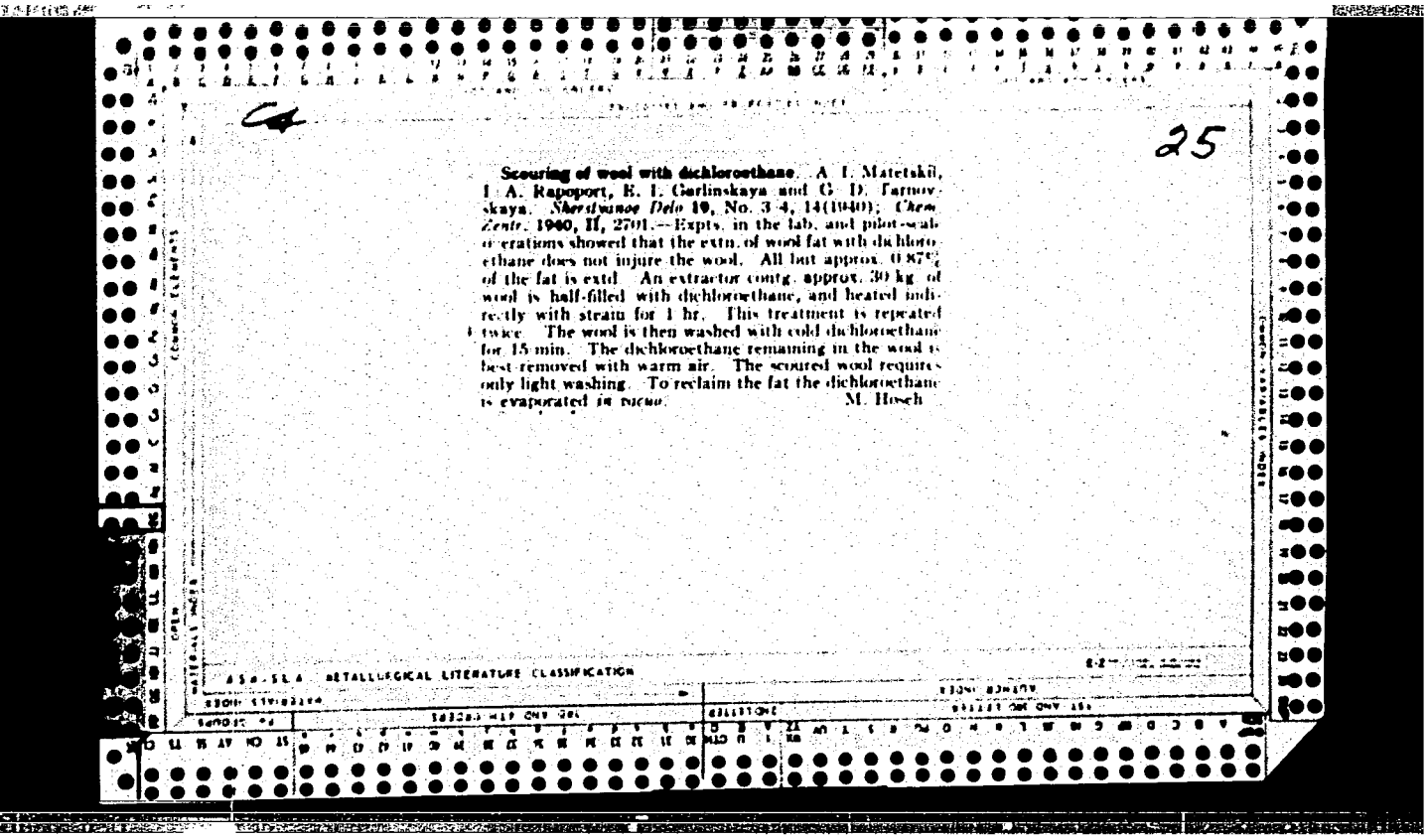


23

Montan wax. Its properties and use in the paper industry. I. A. Rapoport. *Bumazhnaya Prom.* 18, No. 7-8, 1940). — A review of the literature with twenty-three references.
R. Z. Kamich

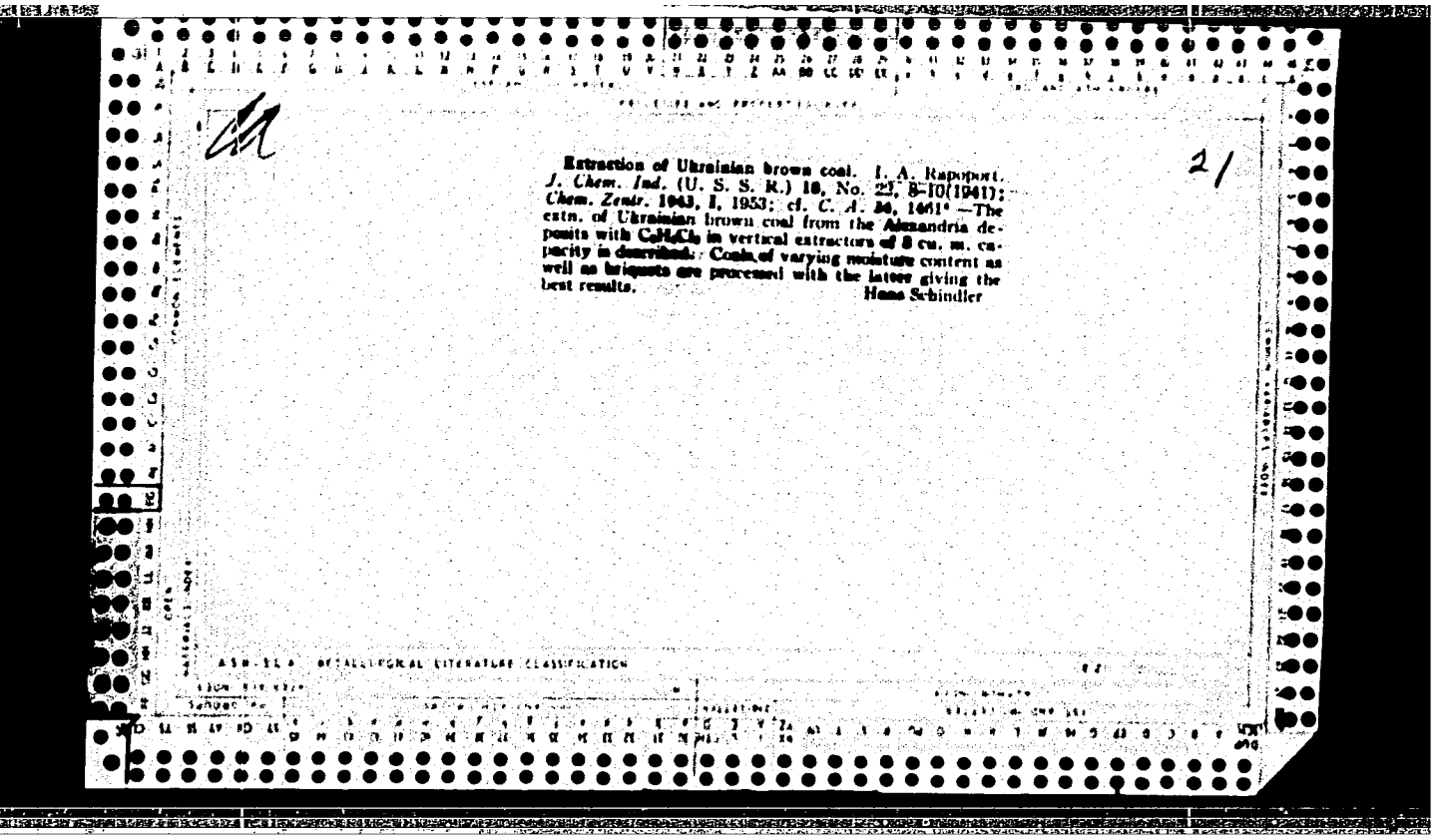


C4

25

Scouring of wool with dichloroethane. A. I. Matetskii, I. A. Rapoport, E. I. Garlinskaya and G. D. Tarnovskaya. *Shersttanoe Delo* **10**, No. 3-4, 14(1940); *Chem. Zentr.* **1940, II**, 2701.—Expts. in the lab. and pilot-scale operations showed that the extn. of wool fat with dichloroethane does not injure the wool. All but approx. 0.87% of the fat is extd. An extractor contg. approx. 30 kg. of wool is half-filled with dichloroethane, and heated indirectly with steam for 1 hr. This treatment is repeated twice. The wool is then washed with cold dichloroethane for 15 min. The dichloroethane remaining in the wool is best removed with warm air. The scoured wool requires only light washing. To reclaim the fat the dichloroethane is evaporated *in vacuo*. M. Hosch

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION



Extraction of Ukrainian brown coal. I. A. Rapoport. *J. Chem. Ind.* (U. S. S. R.) 18, No. 23, 8-10(1941); *Chem. Zentr.* 1943, I, 1953; cf. *C. A.* 36, 1461⁶.—The extn. of Ukrainian brown coal from the Aleksandria deposits with $C_6H_6Cl_6$ in vertical extractors of 3 cu. m. capacity is described. Coals of varying moisture content as well as briquets are processed with the latter giving the best results.

Hans Schindler

1. RAPOPORT, I. A.

2. USSR (600)

4. Waxes

7. Use of technical grade butanol for extraction of montan wax. Ukr. khim. zhur. 18. No. 1, 1952.

9. Monthly List of Russian Accessions, Library of Congress, April 1953. Unclassified.

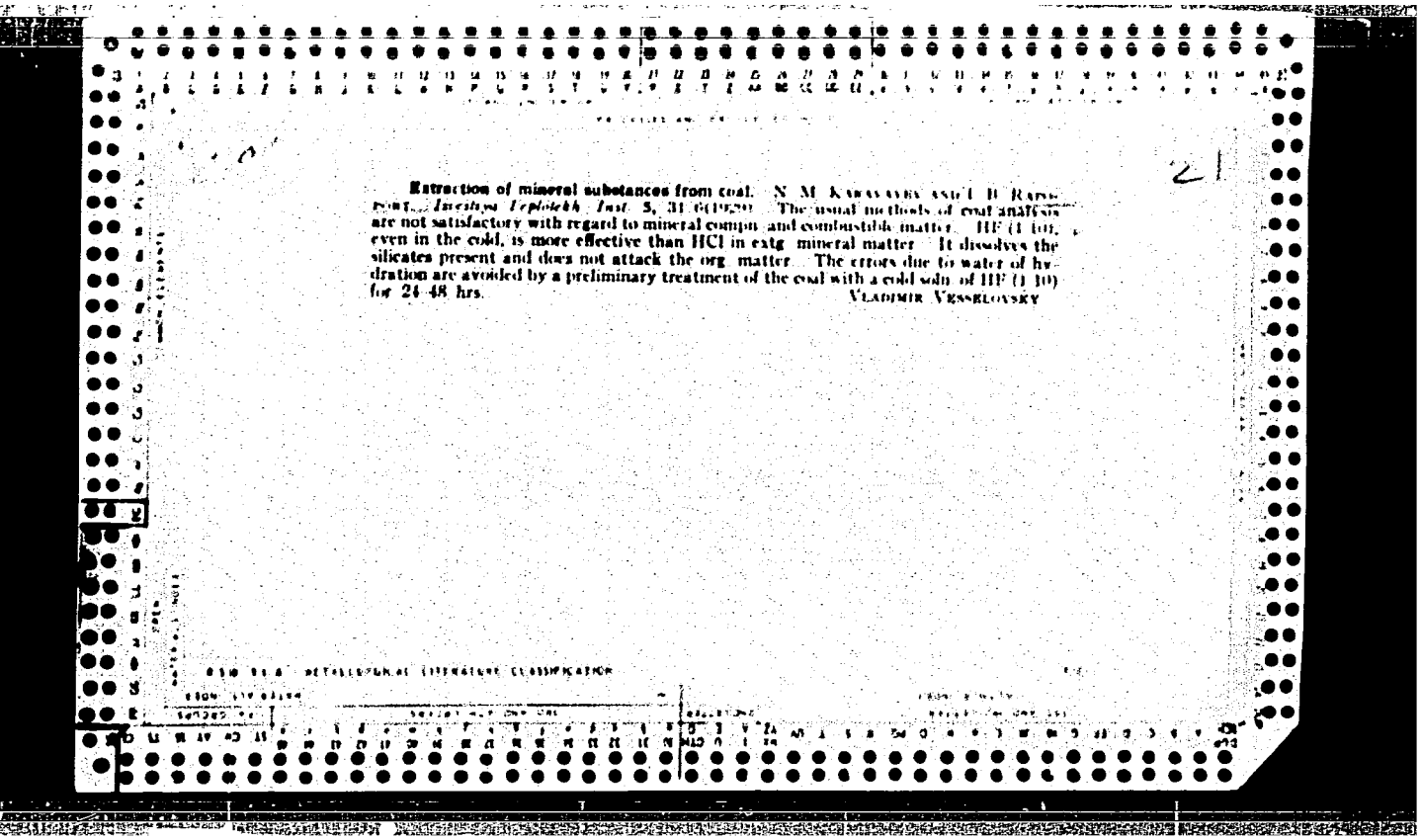
ZASUKHINA, G.D.; RAPOPORT, I.A.

Study of the variability of tick-borne encephalitis complex viruses under the influence of chemical mutagenic factors. Genetika no.5:33-37 N '65. (MIRA 19:1)

1. Institut poliomiyelita i virusnykh entsefalitov AMN SSSR, Moskva i Institut khimicheskoy fiziki AN SSSR, Moskva.

RAPOPORT, I.A.

High mutagenic activity of the nervous inhibitor diisopropyl fluorophosphate. Biul. MOIP. Otd. biol. 68 no.6: 149-153 N-D '63. (MIRA 17:1)

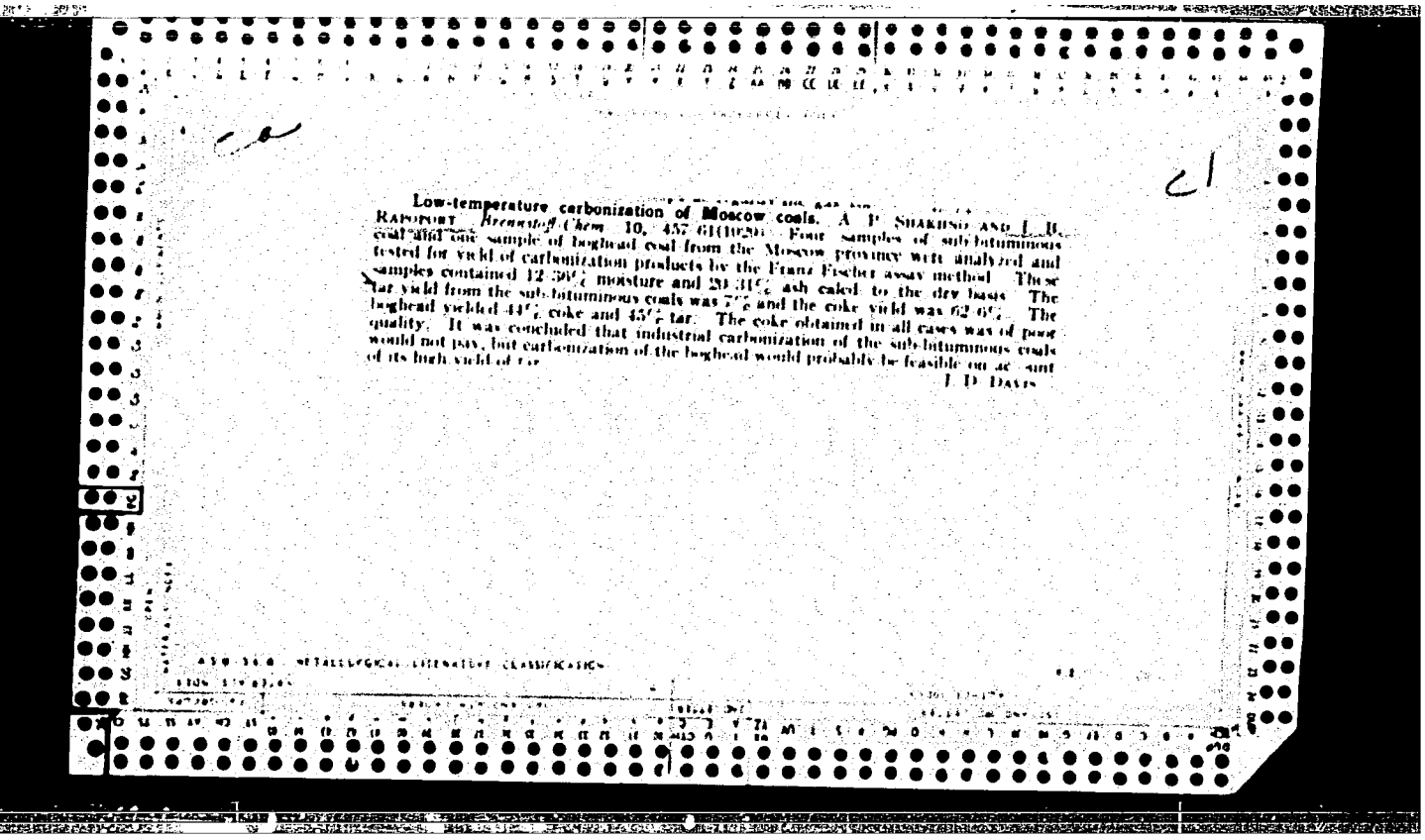


Extraction of mineral substances from coal. N. M. Kamenev and I. D. Rapoport. *Izvestiya Tekhnich. Inst.* 5, 31-6(1929). The usual methods of coal analysis are not satisfactory with regard to mineral compn. and combustible matter. HF (1:10), even in the cold, is more effective than HCl in extg. mineral matter. It dissolves the silicates present and does not attack the org. matter. The errors due to water of hydration are avoided by a preliminary treatment of the coal with a cold soln. of HF (1:10) for 24-48 hrs.

VLADIMIR VESSELOVSKY

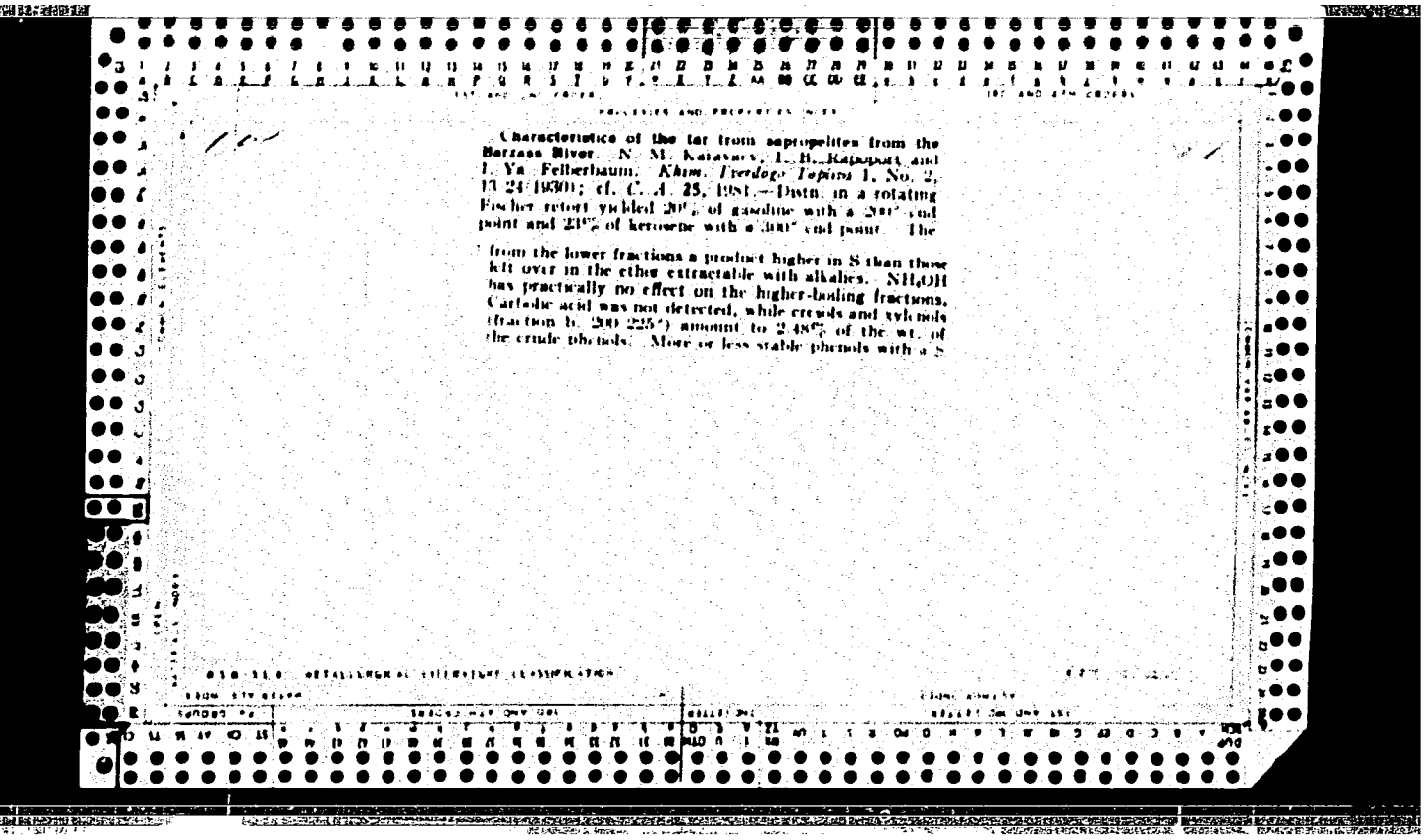
21

Coal from Kuznetski basin (Siberia). N. M. Karavaev and I. B. Rapoport. *Izvestiya Teplotekh. Inst.* 1929, No. 8, 15-30. Coals from the *Lenin* district (Bolduirevski, Serebryanikovskii and Maicrovskii layers) contain about 5% H_2O, while the Zhurinskii layer has up to 10%. The hygroscopic H_2O in the first 3 layers amounts to 1.5-2% and in the fourth to 3.4%. The ash content calcd. on dry coal is 2-11%, S 0.6%, volatile matter 40-45%, and calorific value 7770-8100 cal./kg. The elementary compn. is approx.: C 79.13-84.44, H 5.27-6.05, N 1.05-2.81 and O, in some cases, up to 8%. A low-temp. carbonization carried out in a Fischer retort produced the following results: a baking and swelling semi-coke 67.00-70.74%, gases and loss, 7.61-8.10%, dry tar 13.54-16.60%, moisture in coal 1.98-5.09%, gas 70.3-84.2 cc. per g. coal. The gas had a calorific value of 10,000-11,000 cal./cu. m. and was composed of 8.71-17.80% CO_2, 4.31-10.07% CO, 5.02-6.50% C_nH_{2n}, 58.88-80.02% H_2 + C_nH_{2n+2} and 21.72-21.03% N_2. The tar distillate yielded 6.00-6.35% boiling below 150°, 12.01-15.41% boiling below 200°, 21.52-30.00% boiling below 310° and 40.41-48.00% residue. The 270-340° fraction from the Zhurinskii layer turns to a vaseline-like consistency after a short time, while these fractions (270-340°) are all high in paraffin. Coal from the *Kemerovskii* district contains H_2O 7, ash 1.16-12.90, S 0.6 and volatile matter 21-31%. The calorific value is 8100-8400 cal./kg., while the elementary compn. of the combustible mass averages: C 84.00-85.80, H 4.20-4.00 and N 1.74-1.90%. The low-temp. carbonization produces 80.50-88.84% of baking or a powdered semi-coke, with 3.25-5.90% gases and loss, 4.10-10.35% dry tar, and 2.07-4.28% H_2O. The *Prokopevskii* district coal contains H_2O 5-7, ash 5, S 0.5 and volatile matter 14-22%. The calorific value is 7440-8400 cal./kg. The combustible part is composed of C 86.4-90.0, H 4.1-4.9 and N 1.58-2.47%. A baking or powdery semi-coke amounting to 87.87-92.80% was obtained, while gases and losses were 3.25-4.00%, dry tar 2.14-3.50%, and H_2O 1.84-5.04%. Coal from *Anzhero-Sudzhinskii* district contained moisture 3.3-4.5, ash 3.7-10.2, S 0.6 and volatile matter 14-17%, its calorific capacity was 8680-8800 cal./kg. Its elementary compn. was C 90.6-92.01, H 4.11-4.50 and N 1.6-2.0%. Economical aspects and prices in various parts of the U.S.S.R. are given. A. A. Boehtlingk



Low-temperature carbonization of Moscow coals. A. P. Shakhno and L. B. Rapoport. *Koksnaya i Khim.* 10, 457–61(1939). Four samples of sub-bituminous coal and one sample of boghead coal from the Moscow province were analyzed and tested for yield of carbonization products by the Franz Fischer assay method. These samples contained 12.50% moisture and 20.31% ash calcd. to the dry basis. The tar yield from the sub-bituminous coals was 7% and the coke yield was 62.6%. The boghead yielded 44% coke and 45% tar. The coke obtained in all cases was of poor quality. It was concluded that industrial carbonization of the sub-bituminous coals would not pay, but carbonization of the boghead would probably be feasible on account of its high yield of tar.

J. D. Davis

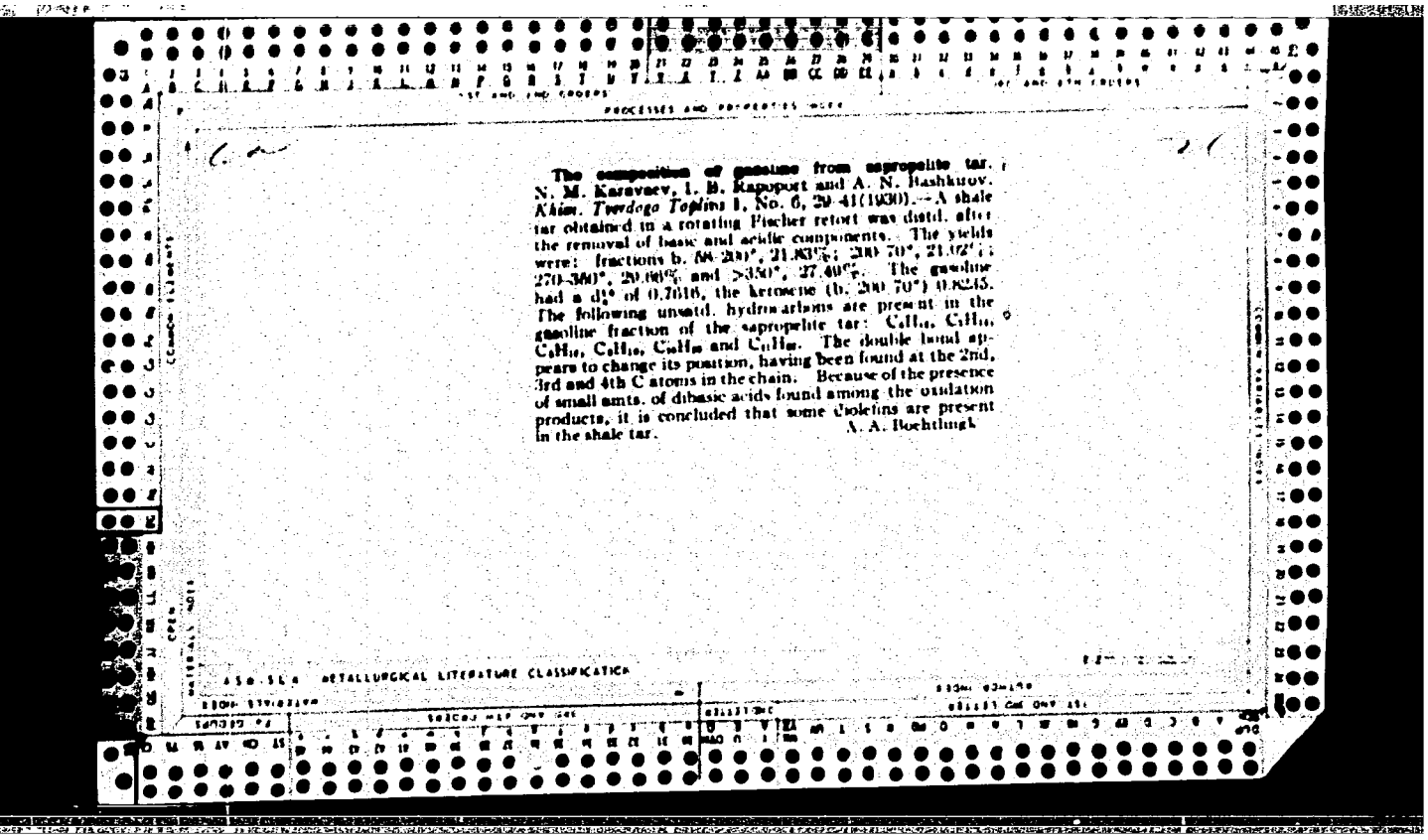


Characteristics of the tar from sapropelites from the Barzass River. N. M. Kanavets, L. B. Rapoport, and I. Ya. Felberbaum. *Khim. Tverdogo Topliva* 1, No. 2, 17-24 (1930); cf. C. A. 25, 1981.—Distn. in a rotating Fischer retort yielded 20% of gasoline with a 200° end point and 21% of kerosene with a 300° end point. The

from the lower fractions a product higher in S than those left over in the ether extractable with alkalies. NH_4OH has practically no effect on the higher-boiling fractions. Carbolic acid was not detected, while cresols and xylenols (fraction b. 200-225°) amount to 2.48% of the wt. of the crude phenols. More or less stable phenols with a S

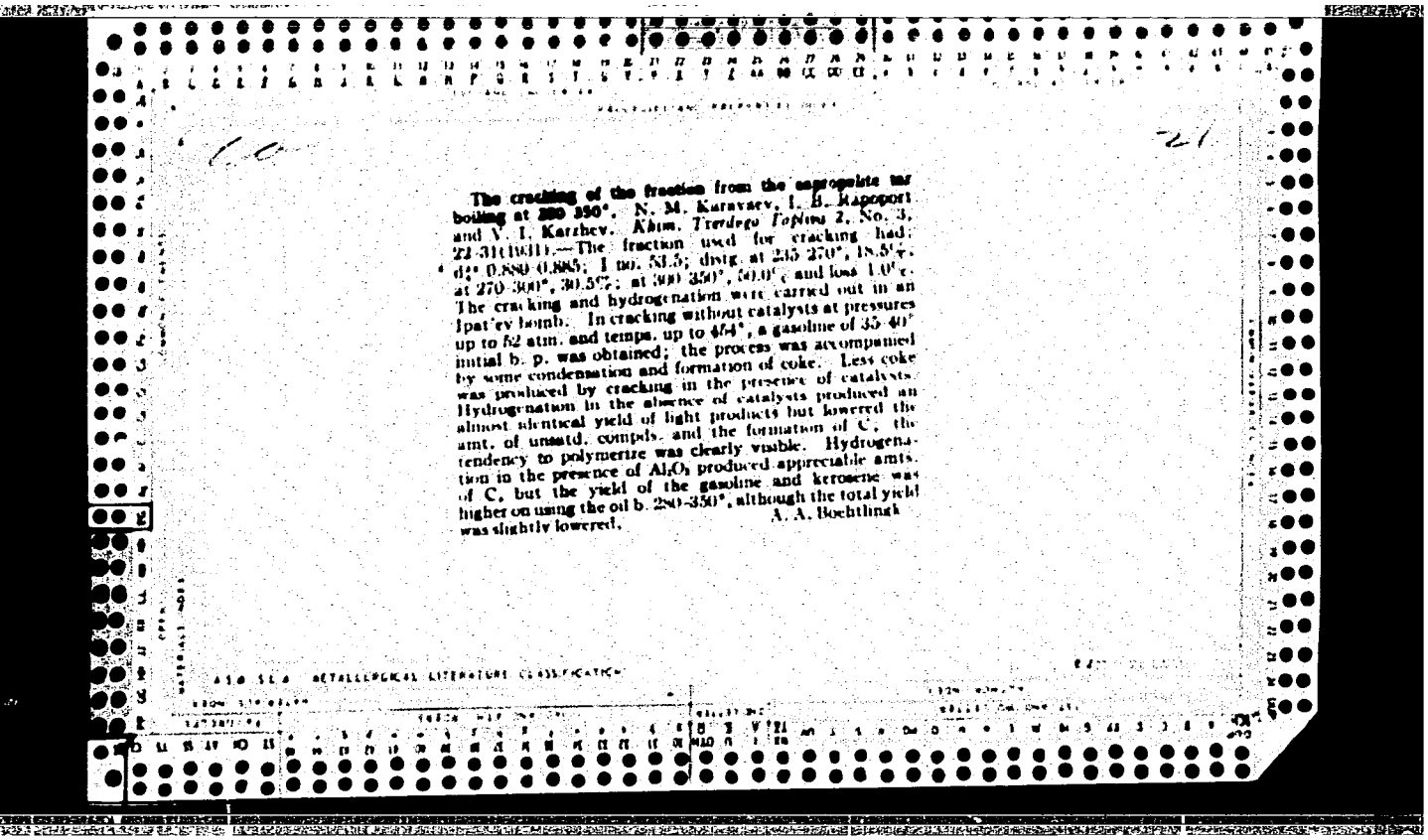
Carboxylic acids from the sapropelite tar from the second deposit of the river Barzass district. H. N. M Karavaev, I. D. Rapoport, and V. A. Kholler. *Khim. Tverdogo Topliva* 1, No. 4, 27-31(1930); cf. C. A. 25, 1881.- The acids obtained on dry-distg. the sapropelites are distributed more or less uniformly in all fractions. Acids from formic up to propionic were found in the tar water; the more complicated ones were extd. from the tar by treating it with a 10% soln. of Na_2CO_3 until the exts. are almost colorless. The exts. are washed with ether to remove neutral compds. and the soln. of the Na salts is treated with a 10% soln. of H_2SO_4 to a strongly acid reaction. The acid layer (upper) is extd. with ether. The aq. soln. is salted out with NaCl and extd. with ether. The ether soln., after drying and evapg. the ether, has a plain-sug. acid odor. The acids had a dark color; 22.5 g. was dissolved in 50 cc. of abs. CH_3OH and esterified with HCl gas, under cooling and for 2 hrs., 20 cc. of CH_3OH was added and HCl was passed in for 1 hr. The Me esters were left for 12 hrs. followed by a careful addn. of Na_2CO_3 to an alk. reaction with litmus. The sepd. esters were extd. with ether, washed with Na_2CO_3 and H_2O and the ether evapd. The remaining alk. soln. was decompd. with 10% H_2SO_4; the ether extn. yielded about 2%, acids which did not take part in the reaction. The obtained Me esters had the following esterification nos.: fraction b. 48-55° 603, 72-79° 475.5, 85-110° 409.5, 130-140° 276.5 and 155-162° 246.0. Phys. characteristics of these fractions are tabulated. The last fraction was vacuum-distd.; at 195-205° a yellow oil was obtained, while the lower-boiling fractions were colorless. Numerous amides also were prepd. and their phys. constants compared with those of known constituents.

A. A. Bachrushin



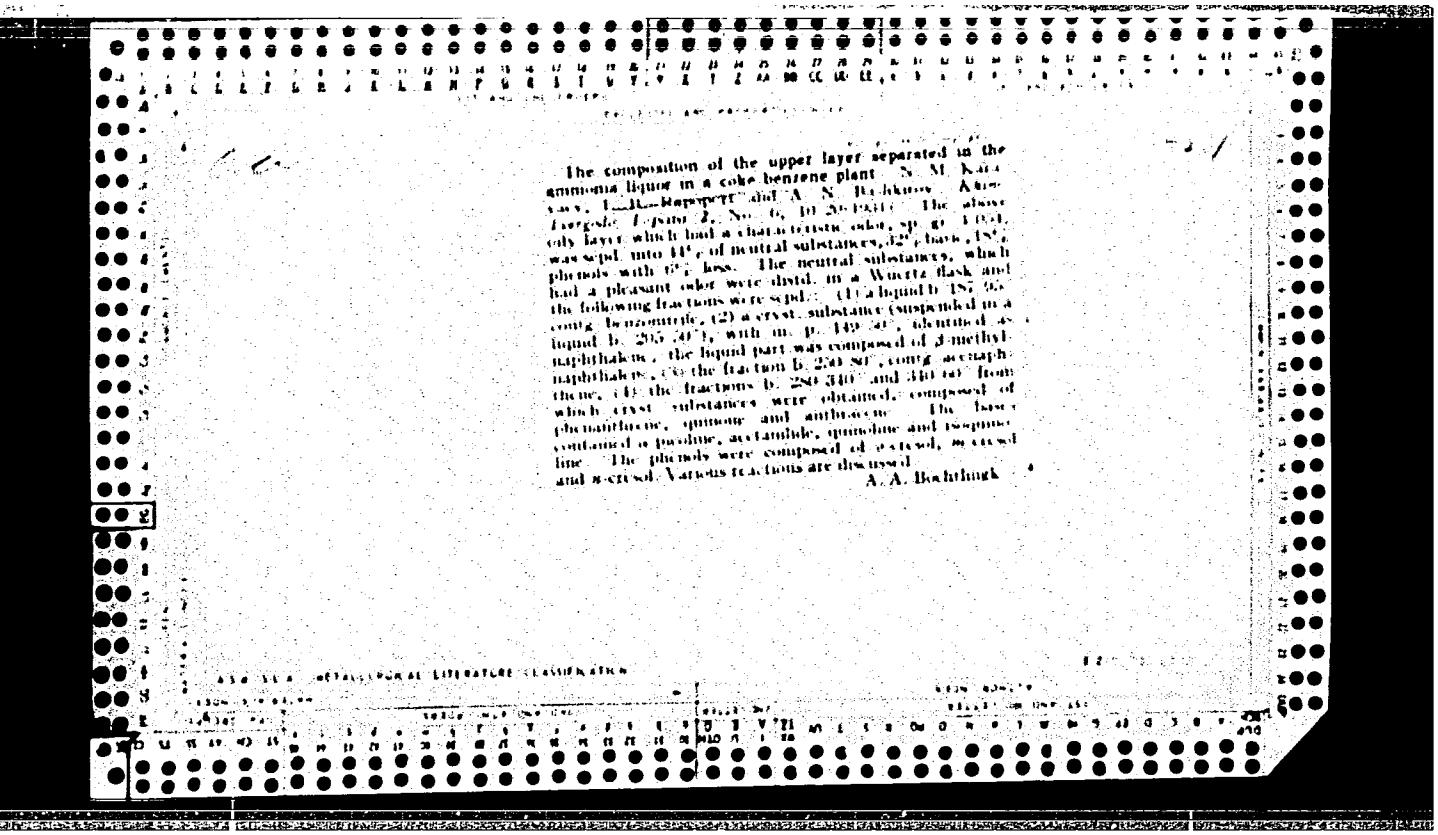
The composition of gasoline from sapropelite tar. N. M. Karavaev, I. B. Rapoport and A. N. Rashkuov. *Khim. Tverdogo Topliva* 1, No. 6, 29-41(1930).—A shale tar obtained in a rotating Fischer retort was distd. after the removal of basic and acidic components. The yields were: fractions b. 60-200°, 21.83%; 200-270°, 21.02%; 270-300°, 20.00%; and >300°, 27.40%. The gasoline had a d_4^{15} of 0.7016, the kerosene (b. 200-270°) 0.8245. The following unsatd. hydrocarbons are present in the gasoline fraction of the sapropelite tar: C_5H_{10}, C_6H_{12}, C_7H_{14}, C_8H_{16} and C_9H_{18}. The double bond appears to change its position, having been found at the 2nd, 3rd and 4th C atoms in the chain. Because of the presence of small amts. of dibasic acids found among the oxidation products, it is concluded that some cholefins are present in the shale tar.

N. A. Boehtlingk



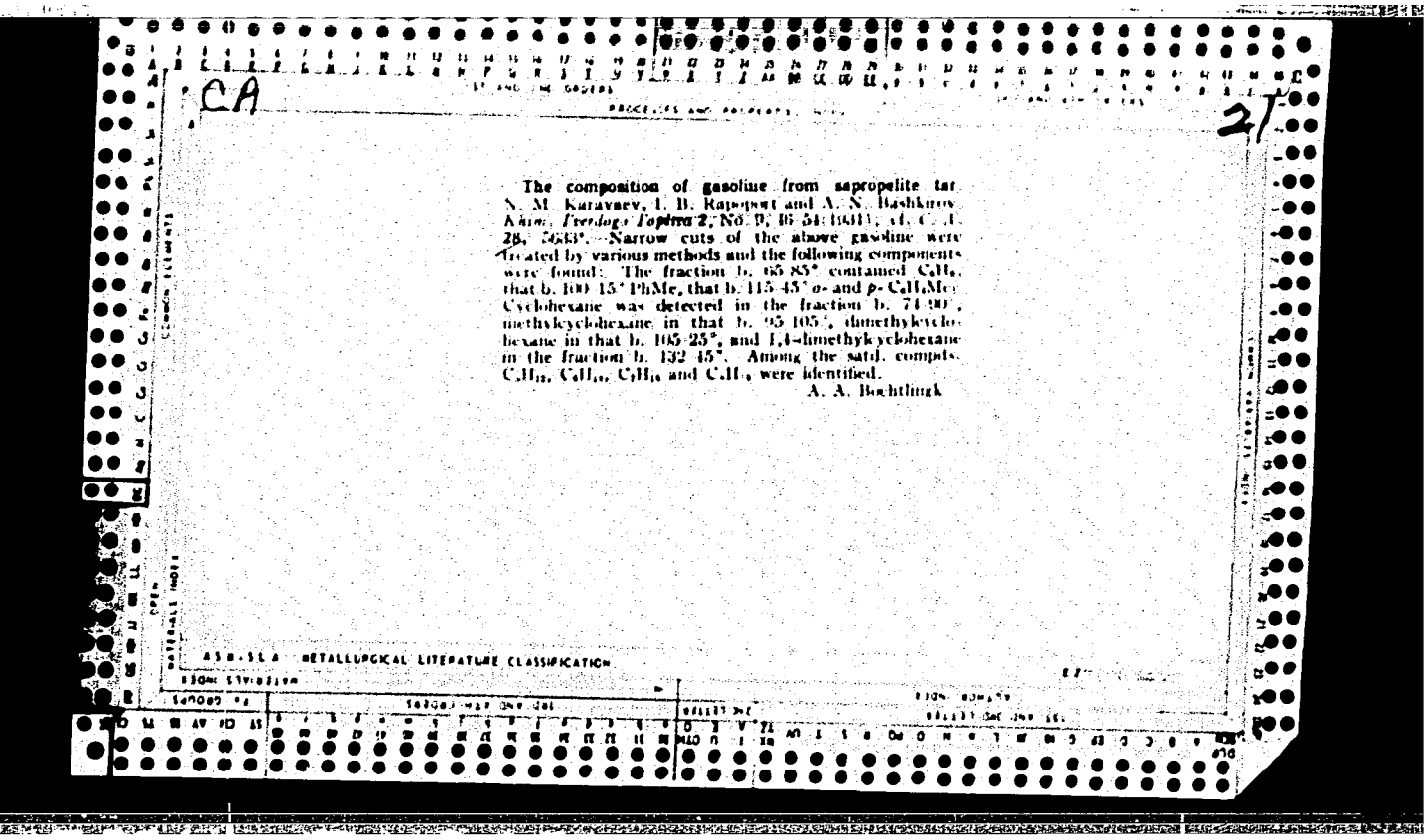
The cracking of the fraction from the naphtenic tar boiling at 280-350°. N. M. Karavaev, I. B. Rapoport and V. I. Karzhev. *Khim. Tverdogo Topliva* 2, No. 3, 22-31(1931).—The fraction used for cracking had d_4^{20} 0.880-0.885; I no. 53.5; distg. at 235-270°, 18.5%; at 270-300°, 30.5%; at 300-350°, 50.0% and loss 1.0%. The cracking and hydrogenation were carried out in an Ipat'ev bomb. In cracking without catalysts at pressures up to 52 atm. and temps. up to 454°, a gasoline of 35-40° initial b. p. was obtained; the process was accompanied by some condensation and formation of coke. Less coke was produced by cracking in the presence of catalysts. Hydrogenation in the absence of catalysts produced an almost identical yield of light products but lowered the amt. of unsatd. compds. and the formation of C, the tendency to polymerize was clearly visible. Hydrogenation in the presence of Al_2O_3 produced appreciable amts. of C, but the yield of the gasoline and kerosene was higher on using the oil b. 280-350°, although the total yield was slightly lowered.

A. A. Boehtlingk



The composition of the upper layer separated in the ammonia liquor in a coke benzene plant. S. M. Kara-vaev, I. L. Rapoport, and A. S. Bachkova. Akademiya Nauk Uzbekskoi S.S.R., Trudy Fiziko-Khimicheskogo Instituta, No. 6, 10-20 (1951). The aqueous layer which had a characteristic odor, sp. gr. 1.034, was separated into 44% of neutral substances, 42% bases, 14% phenols with 6% loss. The neutral substances, which had a pleasant odor were distd. in a Wurtz flask and the following fractions were separated: (1) a liquid b. 187-95° consisting of benzonitrile, (2) a crystalline substance (suspended in a liquid b. 205-40°), with m. p. 149-50°, identified as naphthalene, the liquid part was composed of β-methylnaphthalene, (3) the fraction b. 250-80°, consisting of acenaphthene, (4) the fractions b. 280-340° and 340-60° from which crystalline substances were obtained, composed of phenanthrene, quinone and anthracene. The latter contained a pyridine, acridine, quinoline and isoquinoline. The phenols were composed of o-cresol, m-cresol and p-cresol. Various reactions are discussed.

A. A. Bochkina



CA

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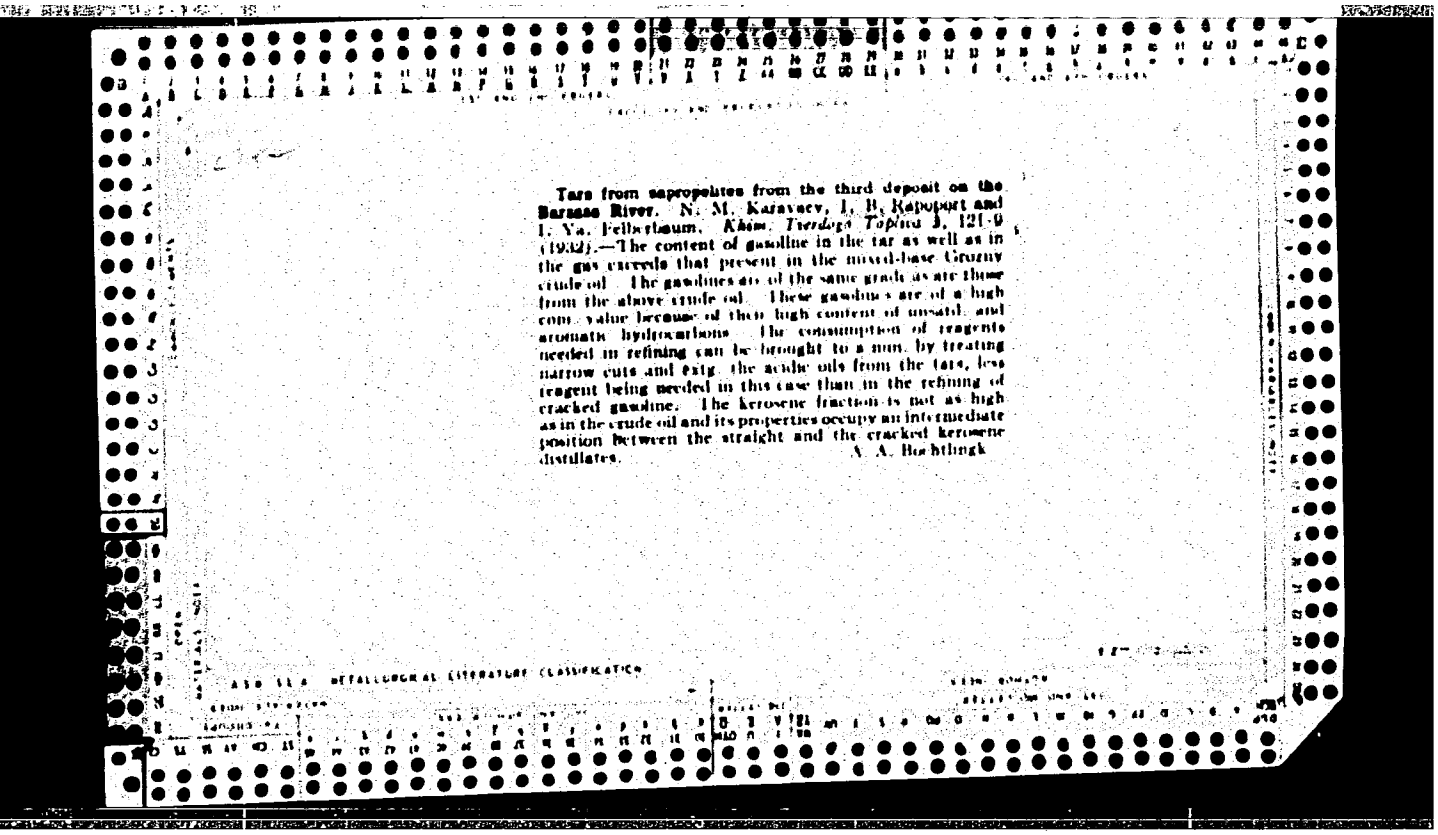
The composition of gasoline from sapropelite tar. N. M. Karavaev, I. B. Rapoport and A. S. Bashkirov. *Khim. Tverdogo Topliva* 2, No. 9, 46-54 (1931); cf. C. A. 28, 5883⁸. Narrow cuts of the above gasoline were treated by various methods and the following components were found: The fraction b. 65-85° contained C_6H_6, that b. 100-15° PhMe, that b. 115-45° *o*- and *p*-$C_6H_4Me_2$. Cyclohexane was detected in the fraction b. 74-80°, methylcyclohexane in that b. 95-105°, dimethylcyclohexane in that b. 105-25°, and 1,4-dimethylcyclohexane in the fraction b. 132-45°. Among the satd. compds. C_nH_{2n}, C_nH_{2n}, C_nH_{2n} and C_nH_{2n} were identified.

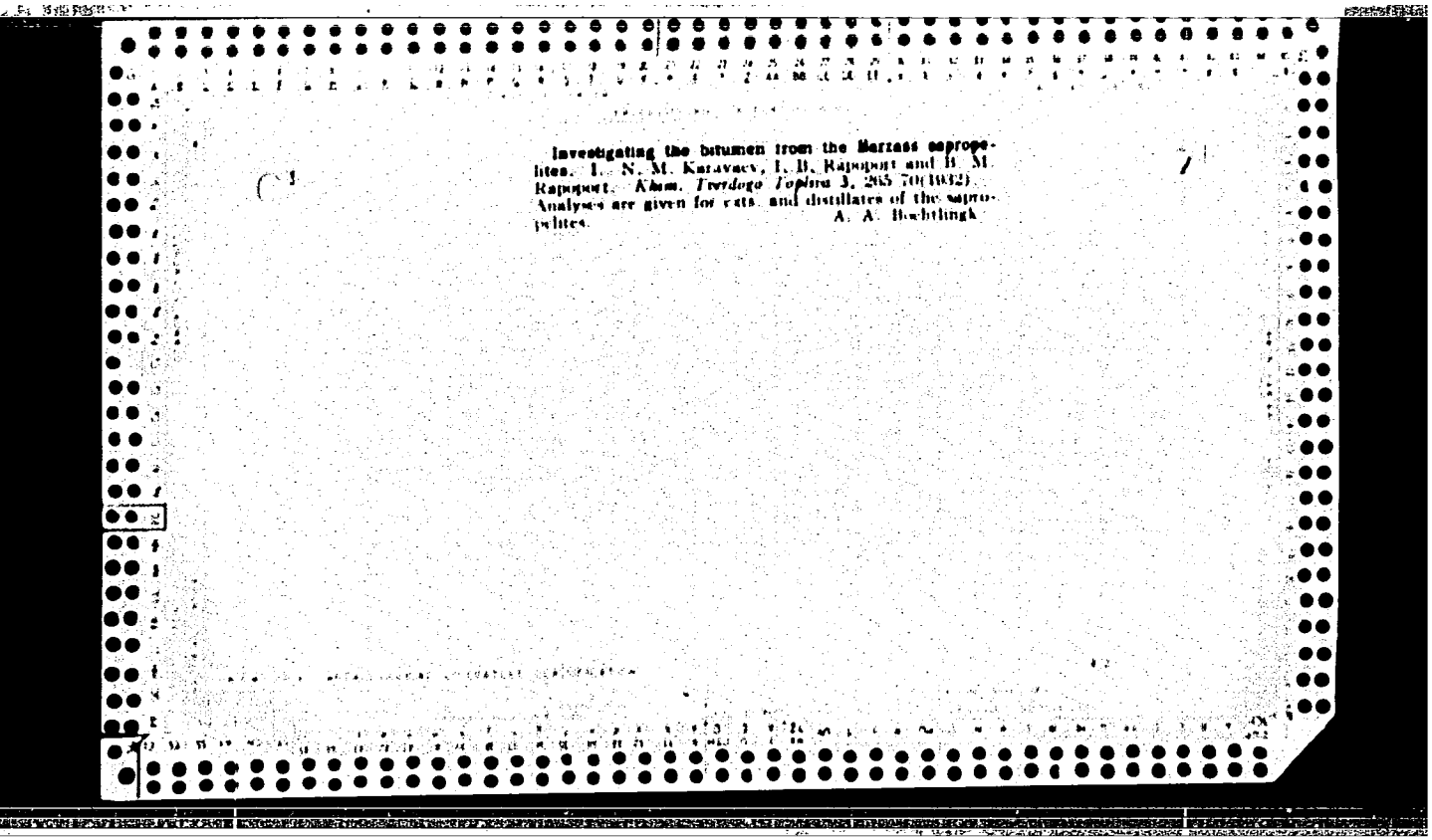
A. A. Boehtlingk

A58-51A METALLURGICAL LITERATURE CLASSIFICATION

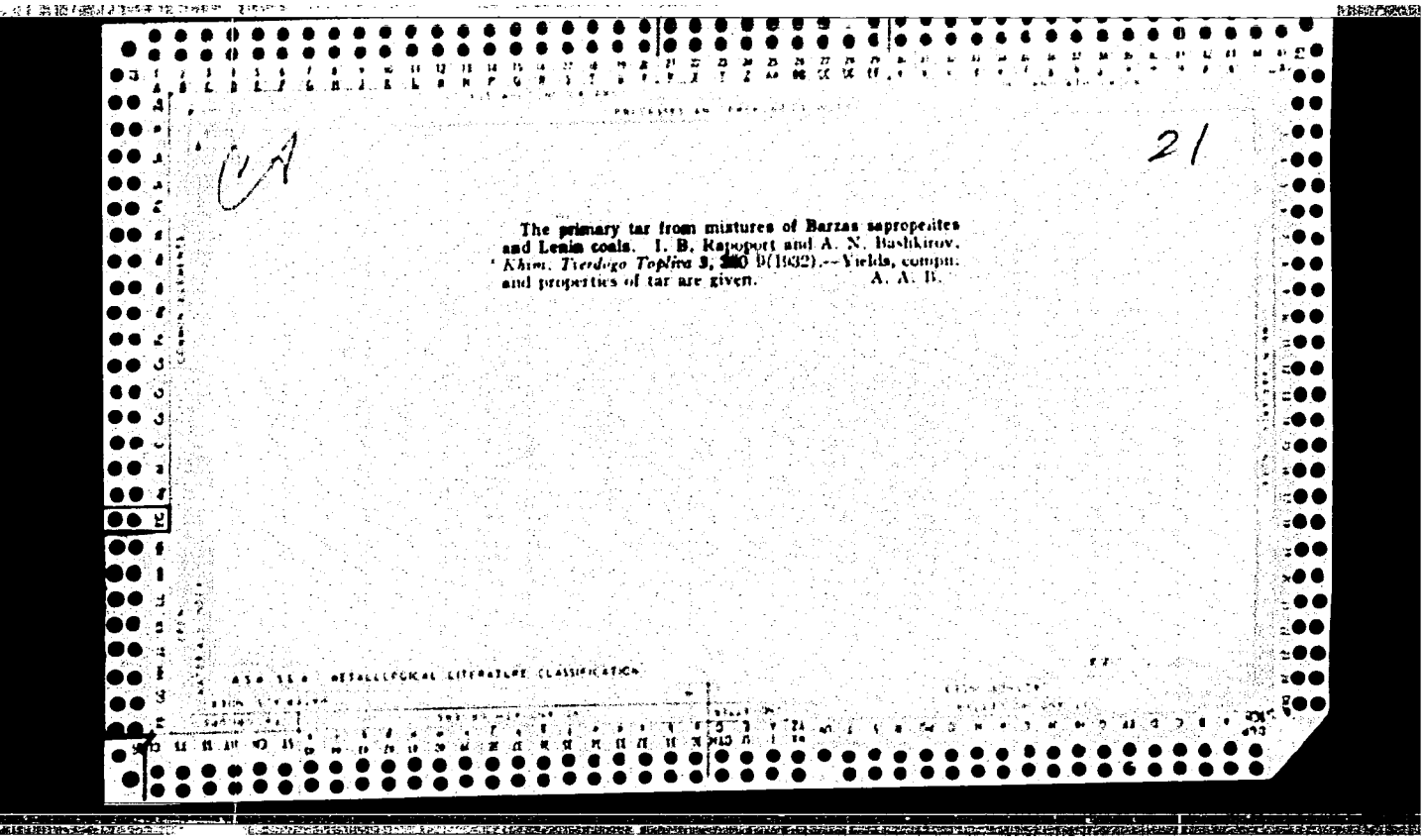


Tars from sapropelites from the third deposit on the Sarmas River. N. M. Karavaev, I. B. Rapoport and I. Ya. Fellerbaum. *Khim. Tverdogo Topliva* 3, 121-9 (1932).—The content of gasoline in the tar as well as in the gas carvels that present in the mixed-base Grozny crude oil. The gasolines are of the same grade as are those from the above crude oil. These gasolines are of a high com. value because of their high content of unsatd. and aromatic hydrocarbons. The consumption of reagents needed in refining can be brought to a min. by treating narrow cuts and extg. the acidic oils from the tars, less reagent being needed in this case than in the refining of cracked gasoline. The kerosene fraction is not as high as in the crude oil and its properties occupy an intermediate position between the straight and the cracked kerosene distillates.

N. A. Hochtlingk



Investigating the bitumen from the Barrass saprope-
lites. I. N. M. Karavaev, L. B. Rapoport and B. M.
Rapoport. *Khim. Tverdogo Topliva* 3, 265-70(1932).
Analyses are given for cuts and distillates of the sapro-
pelites. A. A. Boehtlingk



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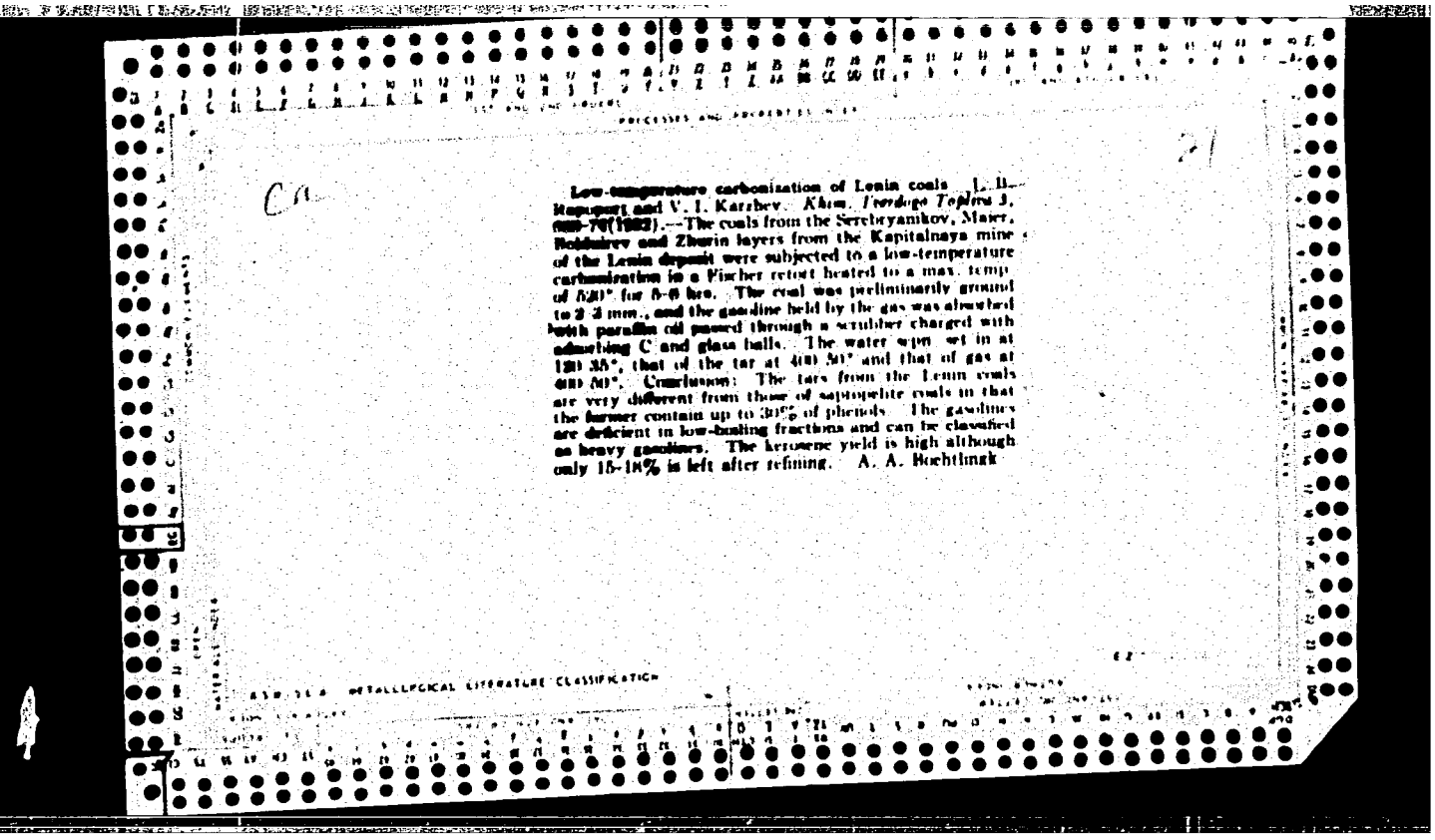
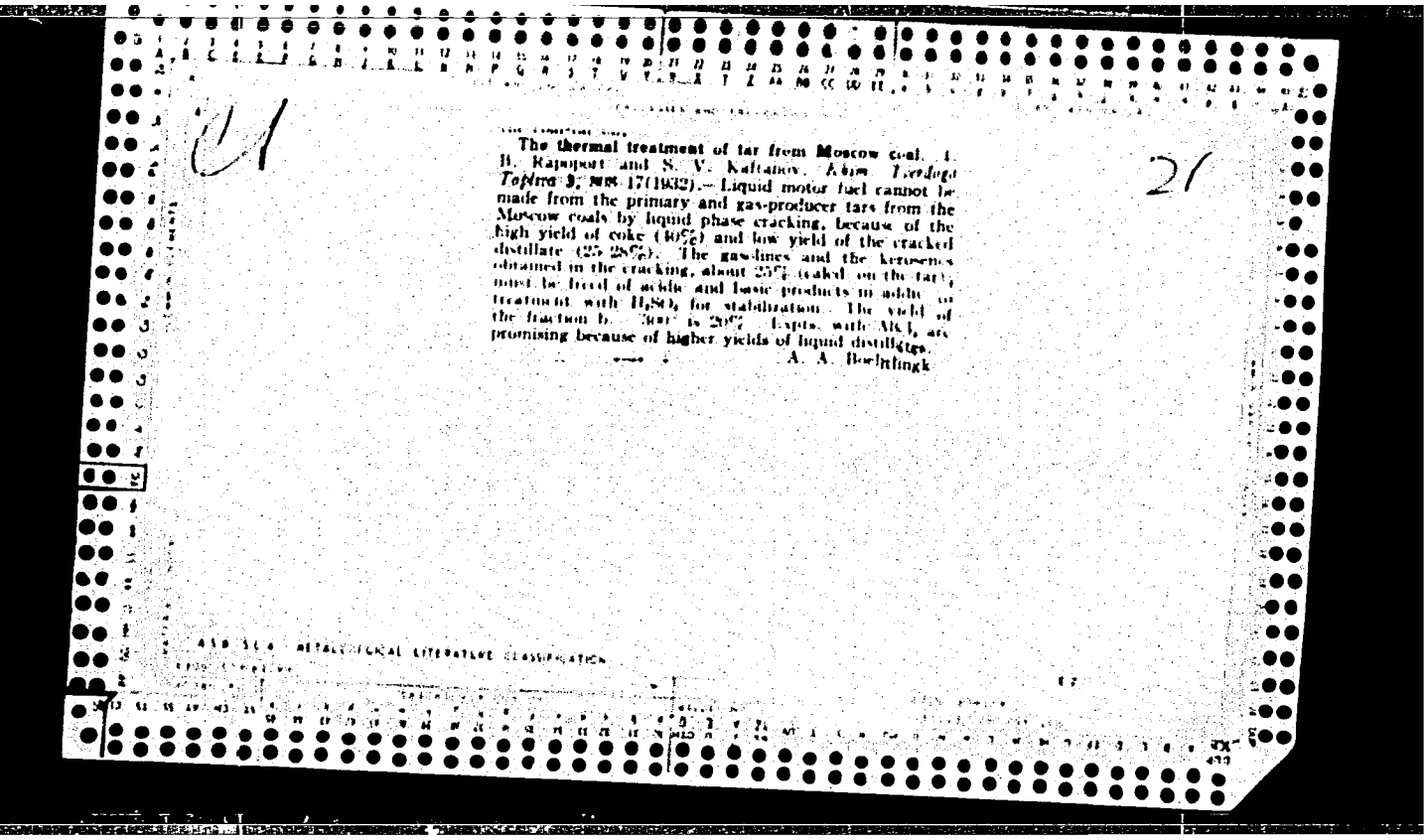
The primary tar from mixtures of Barzas sapropelites and Lenin coals. I. B. Rapoport and A. N. Bashkirov. *Khim. Tverdogo Topliva* 3, 200 9(1932).--Yields, compn. and properties of tar are given. A. A. B.

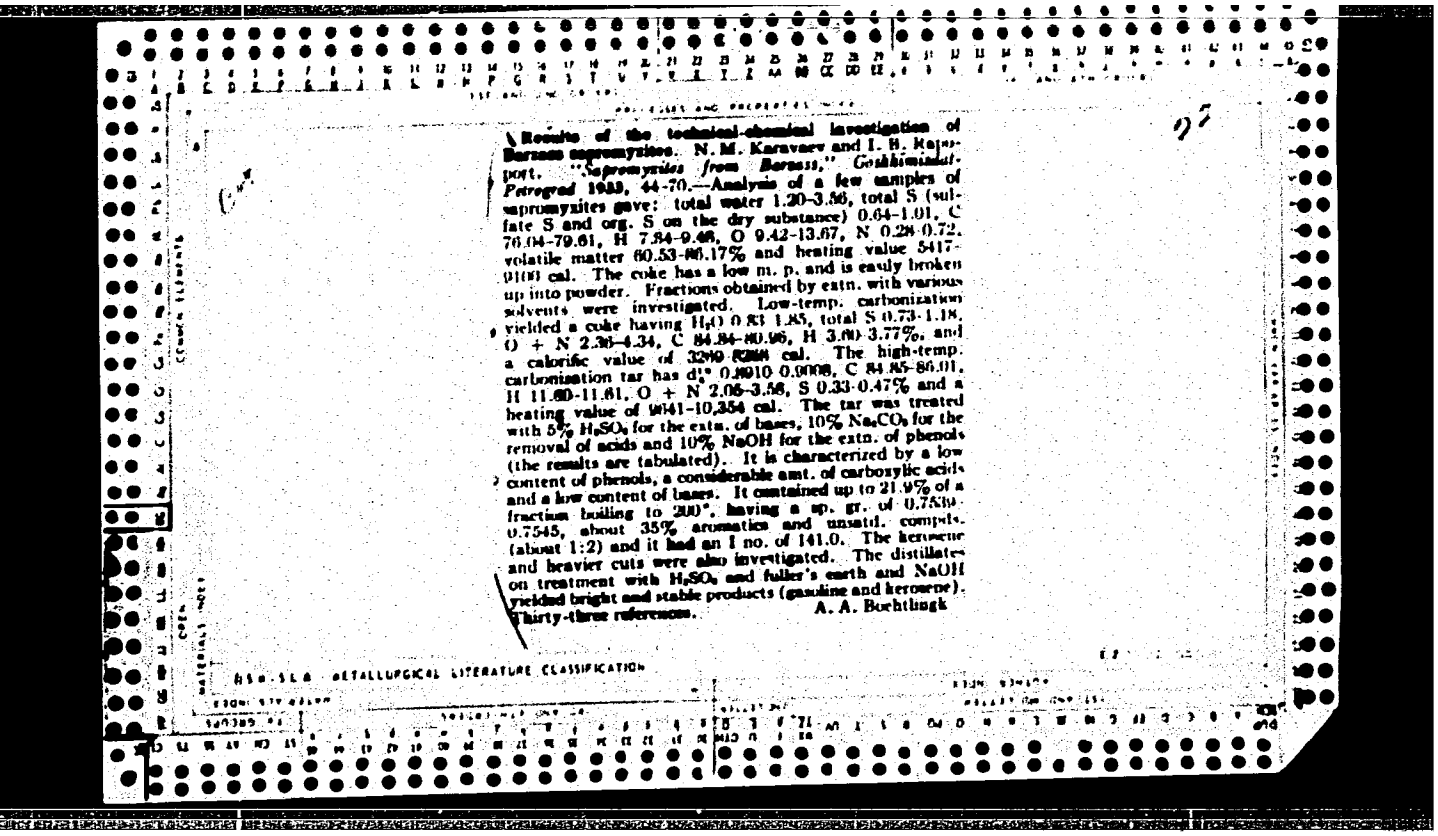


Low-temperature carbonization of Lenin coals. L. B. Rapoport and V. I. Karzhev. *Khim. Tverdogo Topliva* 3, [illegible]-70(1932).—The coals from the Serebryanikov, Maier, Bolshirev and Zhurin layers from the Kapitalnaya mine of the Lenin deposit were subjected to a low-temperature carbonization in a Fischer retort heated to a max. temp. of 520° for 6-8 hrs. The coal was preliminarily ground to 2-3 mm., and the gasoline held by the gas was absorbed with paraffin oil passed through a scrubber charged with adsorbing C and glass balls. The water upon set in at 120-35°, that of the tar at 400-50° and that of gas at 400-50°. Conclusion: The tars from the Lenin coals are very different from those of sapropelite coals in that the former contain up to 30% of phenols. The gasolines are deficient in low-boiling fractions and can be classified as heavy gasolines. The kerosene yield is high although only 15-18% is left after refining. A. A. Bochtlingk



The thermal treatment of tar from Moscow coal. I. B. Rapoport and S. V. Kaftanov. *Khim. Tverdogo Topliva* 3, 808-17(1932).—Liquid motor fuel cannot be made from the primary and gas-producer tars from the Moscow coals by liquid phase cracking, because of the high yield of coke (10%) and low yield of the cracked distillate (25-28%). The gasolines and the kerosenes obtained in the cracking, about 25% (calcd. on the tar), must be freed of acidic and basic products in addn. or treatment with H_2SO_4 for stabilization. The yield of the fraction b. 300° is 20%. Expts. with $AlCl_3$ are promising because of higher yields of liquid distillates.

A. A. Boehlingk

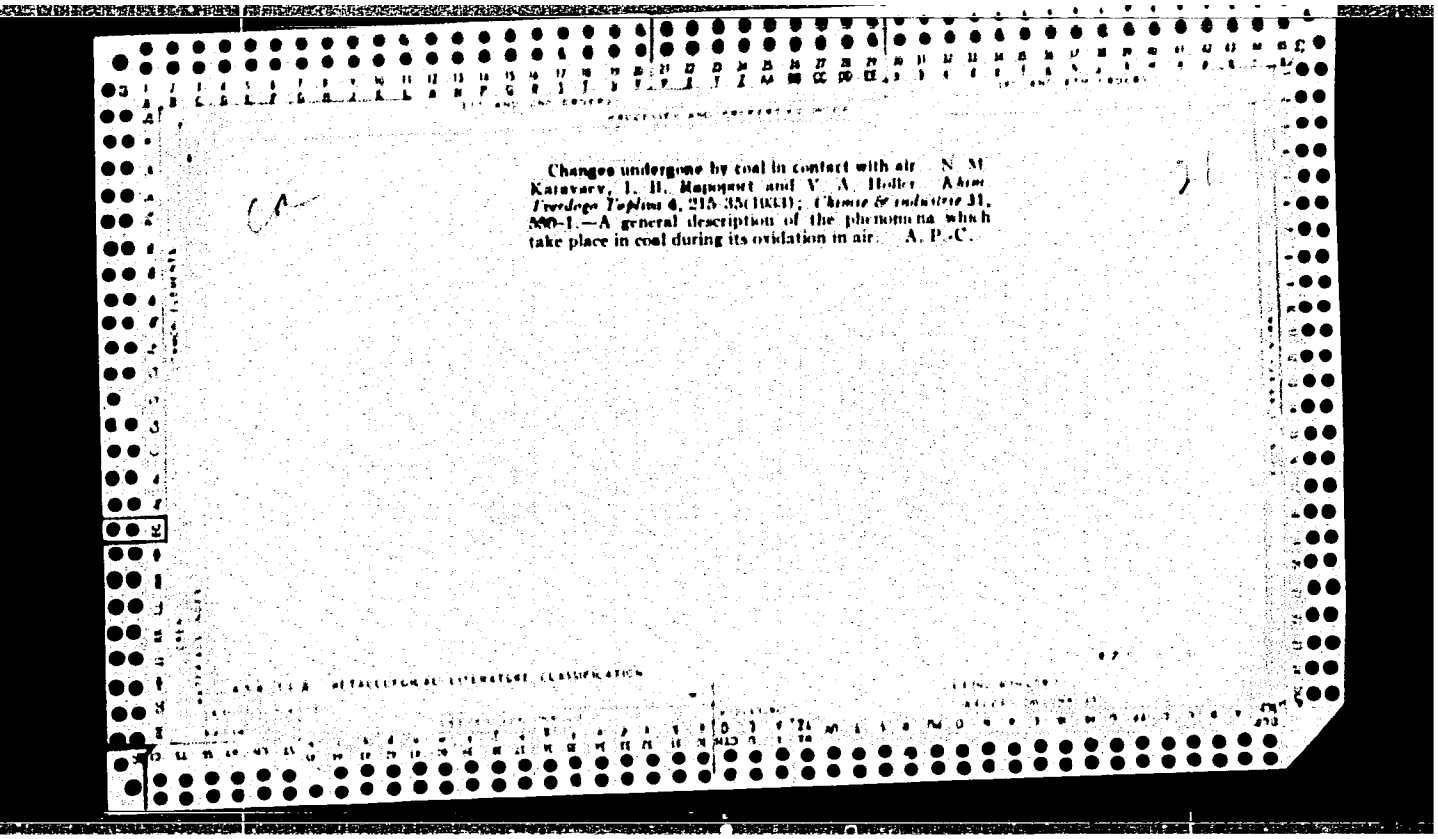


Results of the technical-chemical investigation of Barzass sapromyxites. N. M. Karavaev and I. B. Rapoport. "*Sapromyxites from Barzass*," *Goskhimizdat-Petrograd* 1923, 44-70.—Analysis of a few samples of sapromyxites gave: total water 1.20-3.56, total S (sulfate S and org. S on the dry substance) 0.64-1.01, C 76.04-79.61, H 7.84-9.48, O 9.42-13.67, N 0.28-0.72, volatile matter 60.53-86.17% and heating value 5417-9180 cal. The coke has a low m. p. and is easily broken up into powder. Fractions obtained by extn. with various solvents were investigated. Low-temp. carbonization yielded a coke having H_2O 0.83-1.85, total S 0.73-1.18, O + N 2.30-4.34, C 84.84-80.96, H 3.80-3.77%, and a calorific value of 3249-8268 cal. The high-temp. carbonization tar has d_4^{15} 0.8910-0.9008, C 84.85-86.01, H 11.60-11.61, O + N 2.06-3.58, S 0.33-0.47% and a heating value of 9041-10,354 cal. The tar was treated with 5% H_2SO_4 for the extn. of bases, 10% Na_2CO_3 for the removal of acids and 10% NaOH for the extn. of phenols (the results are tabulated). It is characterized by a low content of phenols, a considerable amt. of carboxylic acids and a low content of bases. It contained up to 21.9% of a fraction boiling to 200°, having a sp. gr. of 0.7539-0.7545, about 35% aromatics and unsatd. compds. (about 1:2) and it had an I no. of 141.0. The kerosene and heavier cuts were also investigated. The distillates on treatment with H_2SO_4 and fuller's earth and NaOH yielded bright and stable products (gasoline and kerosene). Thirty-three references.

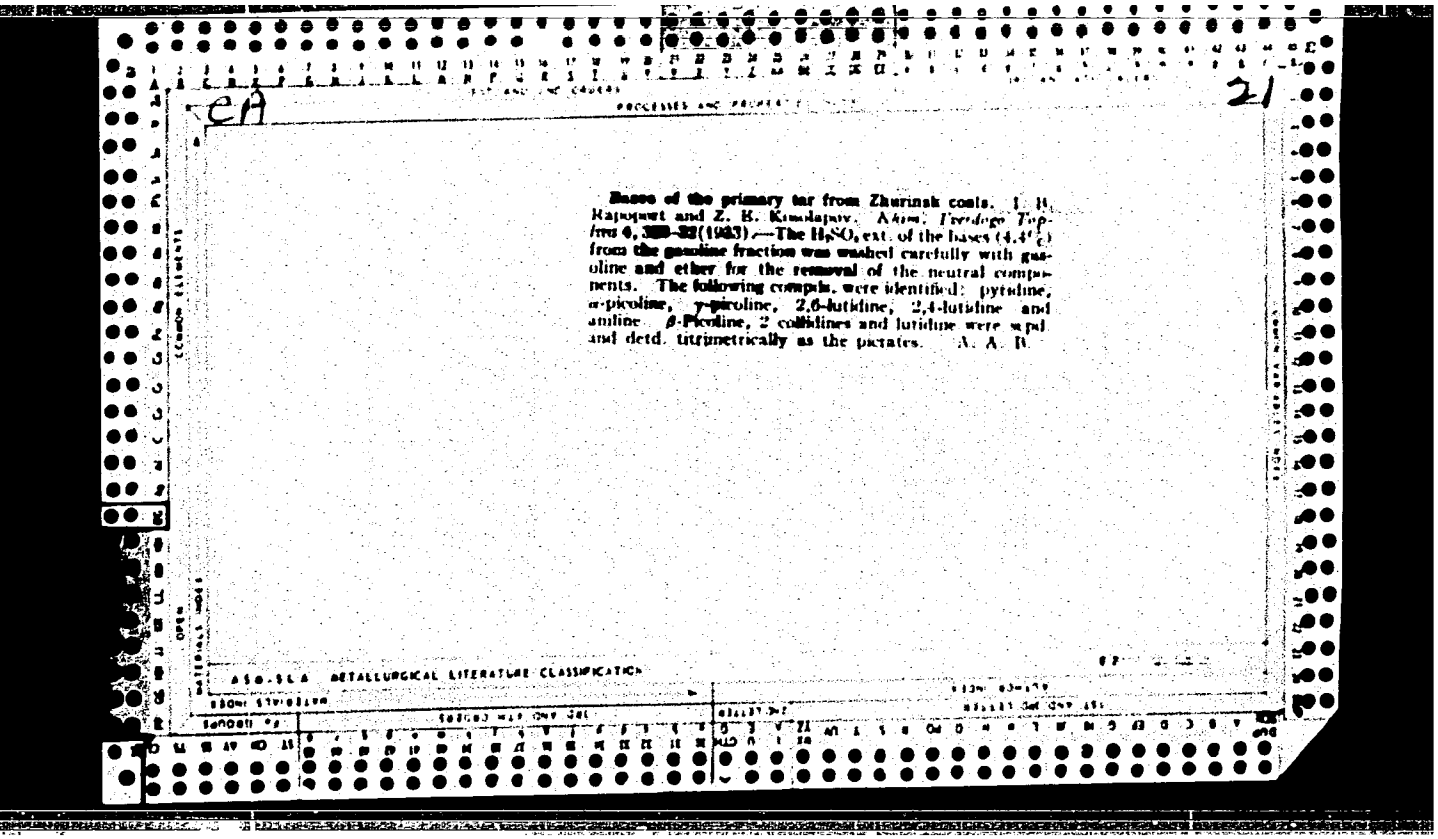
A. A. Boehtlingk

The chemical investigation of low-temperature carbonization products obtained from Dzhanga sapropelites. N. M. Karavaev, I. B. Rabinovich and A. N. Bashkirov. *"Sapropelites from Dzhanga."* *Goskhimizdat-Petrograd* 1933, 91-130.—The low-temp. tar was sepd. into fractions boiling at (1) 28-100°, (2) 92-107°, (3) 115-30°, (4) 130-45°, (5) 145-60°, (6) 160-85°, (7) 185-200°, (8) 200-50°, (9) 250-300°. The following acids were obtained: (1) oleic, valeric and caproic acids, as well as some HCO_2H and AcOH; (2) oleic, valeric and caprylic acids, with traces of HCO_2H and AcOH; (3) oleic, valeric, caproic and enanthic acids, with traces of HCO_2H and AcOH; (4) caproic and enanthic acids and traces of AcOH; (5) valeric, caproic, enanthic and capric acid and traces of AcOH; (6) enanthic and caproic acids; (7) caproic and traces of HCO_2H. (8) could not be properly analyzed and the main treated portion had a b. p. of 98-125°. (9) yielded a product b. 120-52°. The following unsatd. hydrocarbons were found: C_6H_{12}, C_7H_{14}, C_8H_{16}, C_9H_{18}, $C_{10}H_{20}$, $C_{11}H_{22}$, $C_{12}H_{24}$, $C_{13}H_{26}$, $C_{14}H_{28}$ and $C_{15}H_{30}$. The double bond was at the 2nd, 3rd or 4th C atom. A small amt. of diolefins was also traced. The aromatic compds. were investigated in the fractions: (1) 30-100°, (2) 100-15°, (3) 115-45°, (4) 150-6°, (5) 156-61°, (6) 161-7°, (7) 167-75°, (8) 175-85° and (9) 185-210°. Fraction (1) (b. 65-85°) contained C_6H_6; (2) toluene; (3) *o*- and *p*-xylene; (4) mesitylene and pseudocumene; (5) mesitylene, pseudocumene and small amts. of *p*-ethyltoluene; (6) mesitylene and pseudocumene, (7) pseudocumene and hemimellitene; (8) hemimellitene; and (9) isodurene and prehnitene. The following naphthenes were found: fraction b. below 60° cyclohexane, 95-105° methylcyclohexane, 132-145° 1,4-dimethylcyclohexane. The following paraffins were found in the fraction b. 35-125.5°: C_6H_{14}, C_7H_{16}, C_8H_{18} and C_9H_{20}. The following acids were isolated from 5 fractions b. 48-162°: caproic, enanthic, caprylic, pelargonic, capric, undecylic, lauric, tridecoic and myristic. The 280-350° fraction on being cracked yielded up to 32% of gasoline and 52-4% kerosene at an operating pressure of about 60 atm. and a temp. of 450°. Hydrogenation in the presence of Al_2O_3 at 70-230 atm. for 4 hrs. at 415-25° yielded 22.4% of a fraction b. below 170°, 11% 170-230°, 9.65% 230-80° and 3.57% at 280-350°, and was accompanied by coke formation. Hydrogenation expts. carried out in the presence of Al_2O_3 + CuO and Al_2O_3 + (Al_2O_3 + CuO) at 440-55° and 405-30° are also reported. Heavy bottoms b. above 350° were hydrogenated in the presence and absence of the above catalysts. The gasoline and kerosene fractions obtained are compared with those from petroleum products. In the hydrogenation of the sapropelite in the presence of Co oxide and NH_4 molybdate at 430-450° in a rotating Bergius autoclave the yield after about 1 hr. and 40 min. of oily products reached 40-3.5% and that of the solid residue 38-43.2%, while the gaseous part was composed mainly of C_nH_{2n+2} hydrocarbons. A great variety of hydrogenation expts. are reported. Conclusion: The sapropelite tars are high in unsatd. hydrocarbons (chiefly iso forms), and they also contain aromatic compds. and naphthenes and 40-60% satd. compds.

A. A. Boehtlingk



Changes undergone by coal in contact with air. N. M. Karavaev, I. B. Rapoport and V. A. Holler. *Khim. Tverdogo Topliva* 6, 215-35(1935); *Chimie & industrie* 31, 800-1.—A general description of the phenomena which take place in coal during its oxidation in air. A. P.-C.

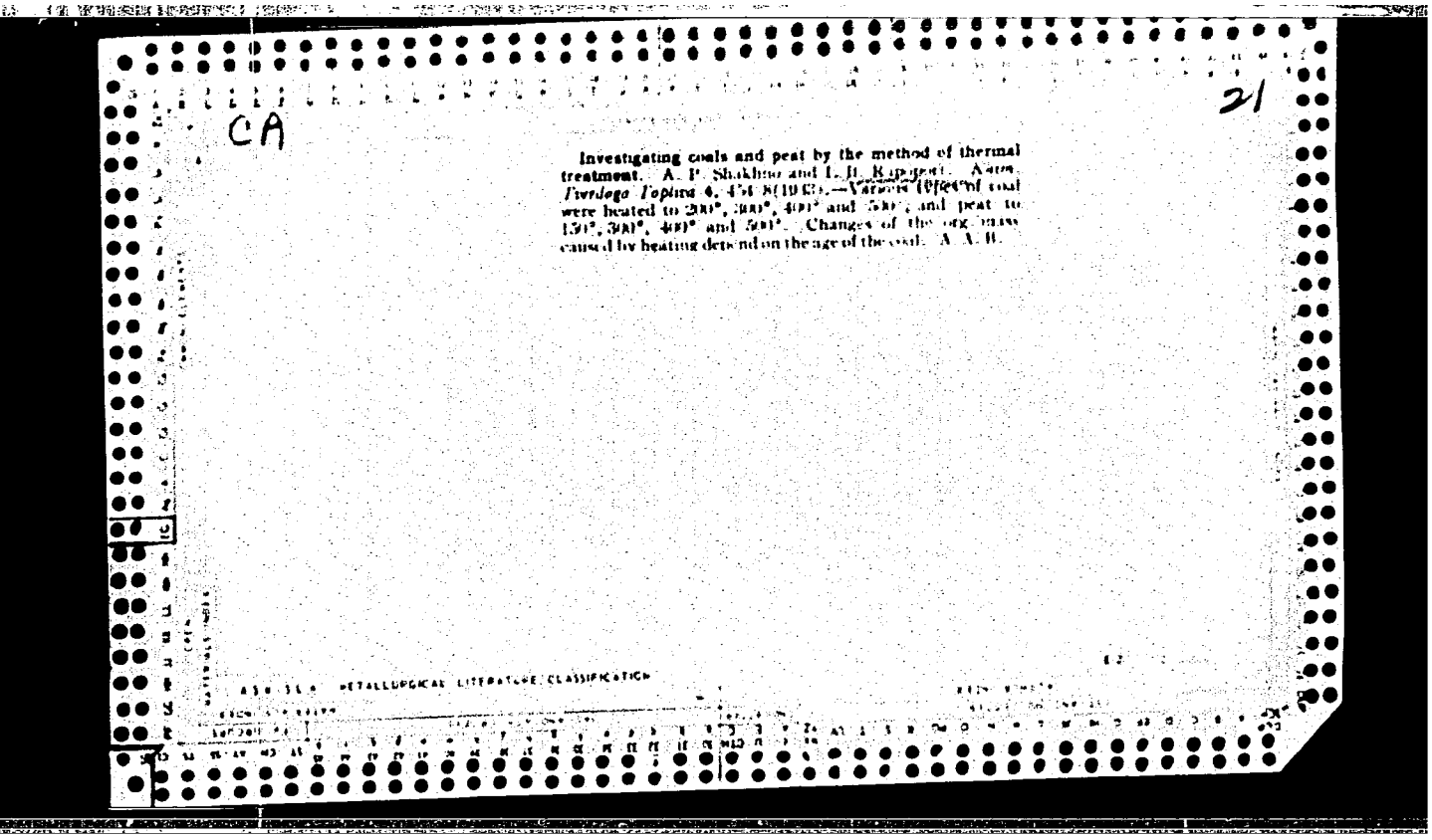


CA

21

Bases of the primary tar from Zhurinsk coals. I. B. Rapoport and Z. B. Kimdapov. *Khim. Tverdogo Topliva* 6, 388–92(1983).—The H_2SO_4 ext. of the bases (4.4%) from the gasoline fraction was washed carefully with gasoline and ether for the removal of the neutral components. The following compds. were identified: pyridine, α-picoline, γ-picoline, 2,6-lutidine, 2,4-lutidine and aniline. β-Picoline, 2 collidines and lutidine were sepd. and detd. titrimetrically as the picrates. A. A. B.

ASD-SLA METALLURGICAL LITERATURE CLASSIFICATION



CA

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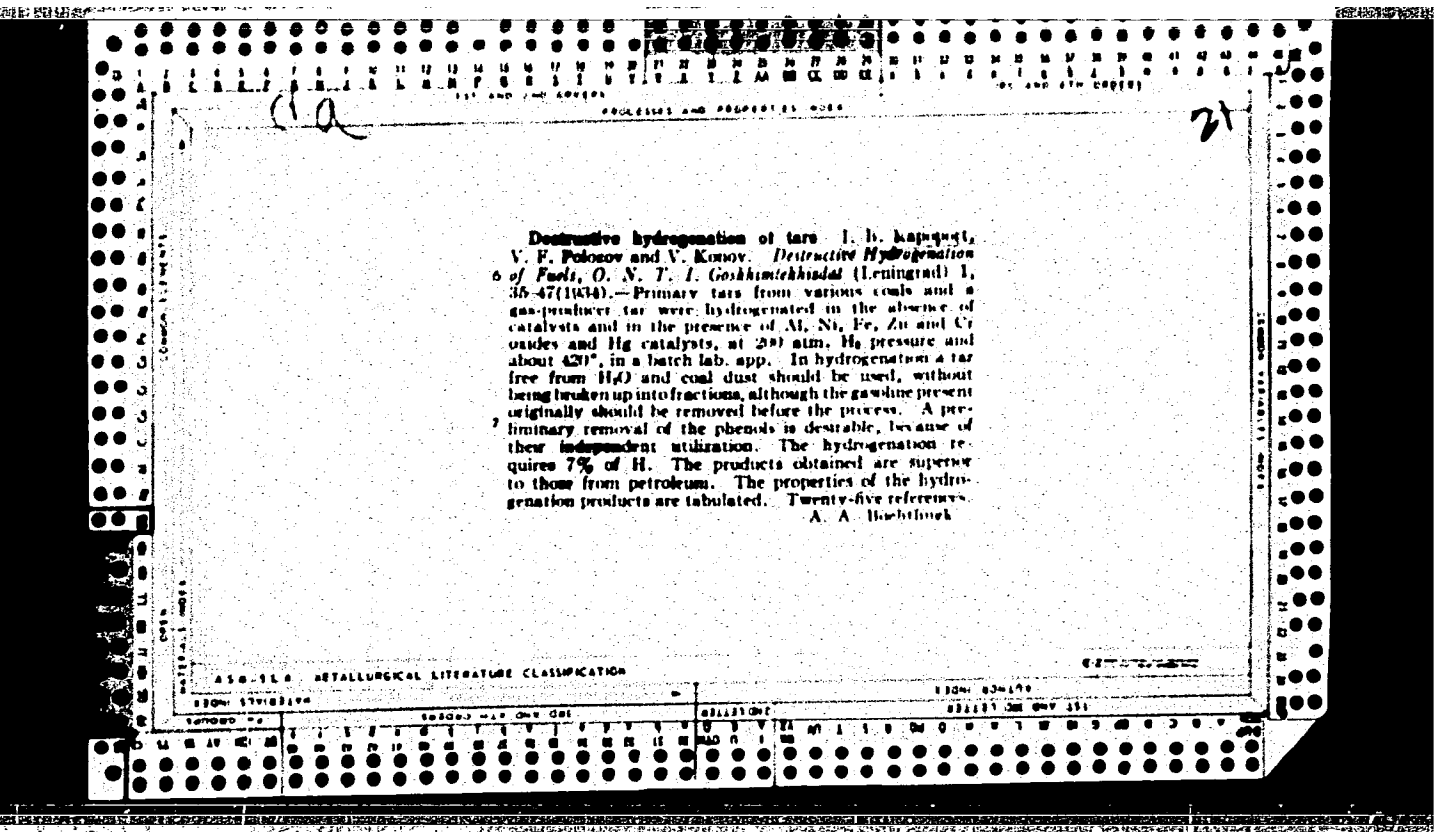
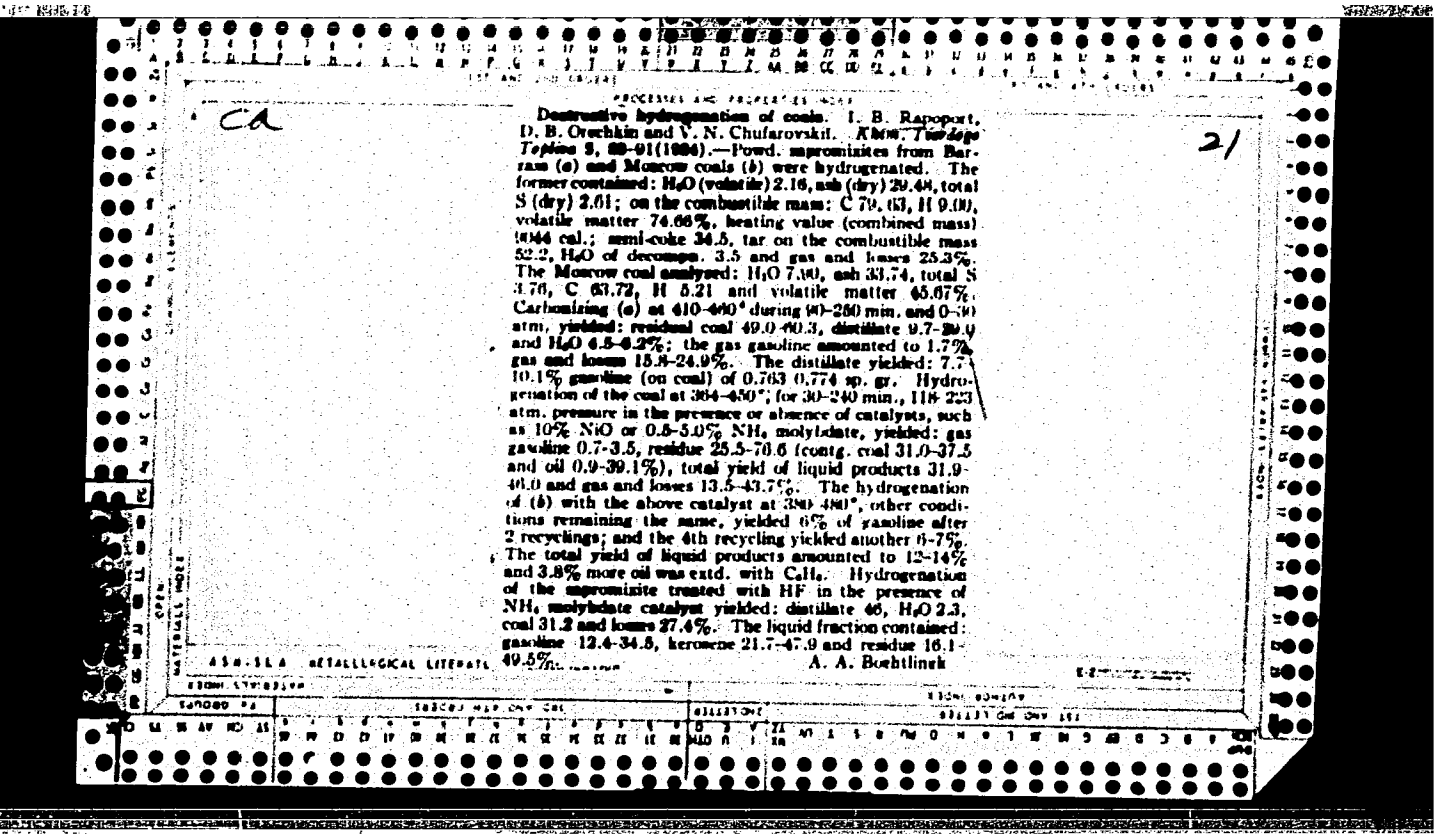
Investigating coals and peat by the method of thermal treatment. A. P. Shakhov and L. [illegible] Kapgent. Acta. *Tverdogo Topliva* 6, 451-8(1935).—Various types of coal were heated to 200°, 300°, 400° and 500°, and peat to 150°, 300°, 400° and 500°. Changes of the org. mass caused by heating depended on the age of the coal. A. A. B.

A 5 5 1 A METALLURGICAL LITERATURE CLASSIFICATION



Destructive hydrogenation of tars I. B. Kapustt, V. F. Poloxov and V. Kuxov. *Destructive Hydrogenation of Fuels, O. N. T. I. Goskhimtekhizdat* (Leningrad) 1, 35-47(1938).—Primary tars from various coals and a gas-producer tar were hydrogenated in the absence of catalysts and in the presence of Al, Ni, Fe, Zn and Cr oxides and Hg catalysts, at 200 atm. H₂ pressure and about 420°, in a batch lab. app. In hydrogenation a tar free from H_2O and coal dust should be used, without being broken up into fractions, although the gasoline present originally should be removed before the process. A preliminary removal of the phenols is desirable, because of their independent utilization. The hydrogenation requires 7% of H. The products obtained are superior to those from petroleum. The properties of the hydrogenation products are tabulated. Twenty-five references.

A. A. Baikhirsch



CA

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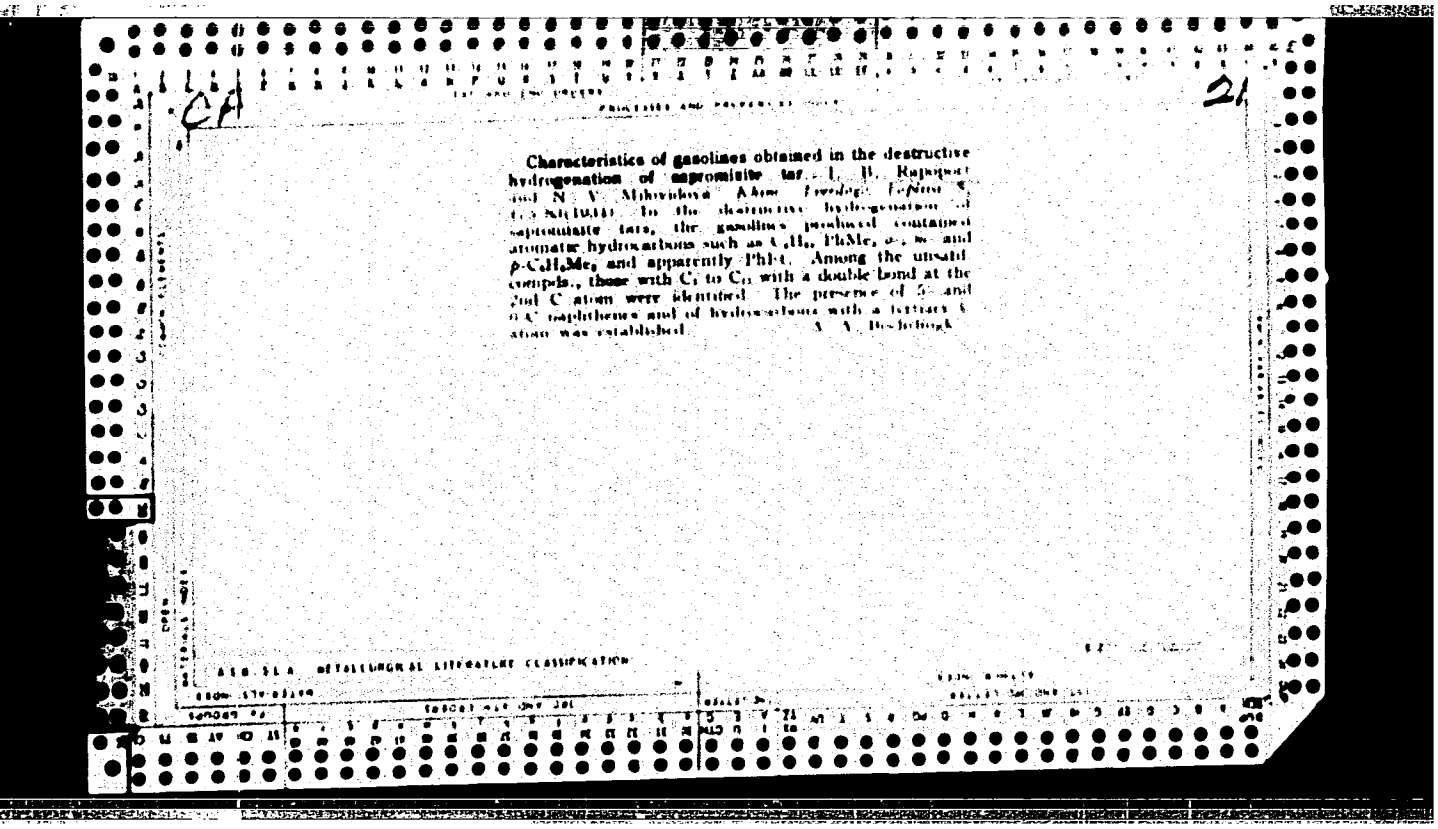
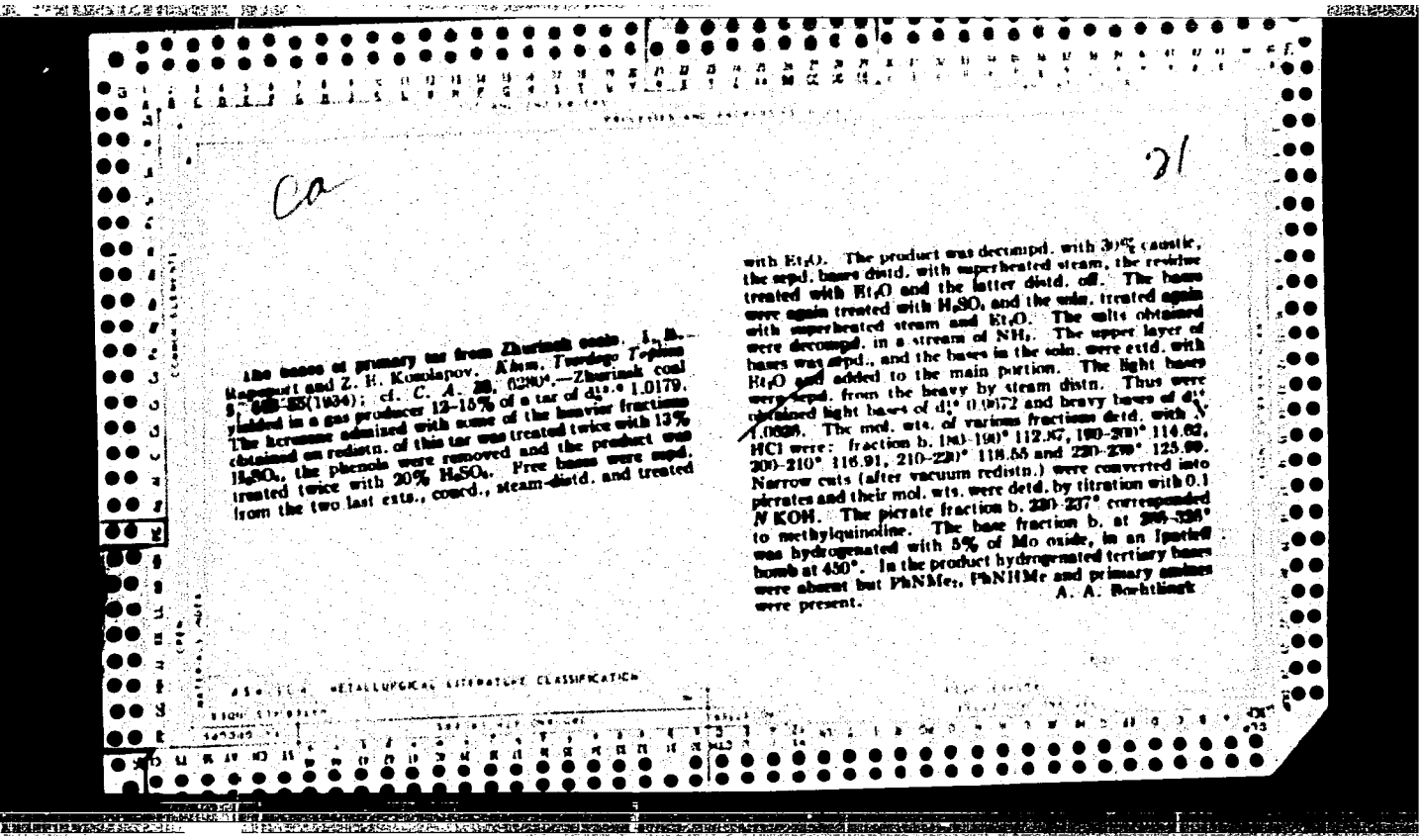
Destructive hydrogenation of coals. I. B. Rapoport, D. B. Orekhkin and V. N. Chufarovskii. *Khim. Tverdogo Topliva* **5**, 89–91(1934).—Powd. supromixites from Barrau (*a*) and Moscow coals (*b*) were hydrogenated. The former contained: H_2O (volatile) 2.16, ash (dry) 29.48, total S (dry) 2.61; on the combustible mass: C 79.63, H 9.00, volatile matter 74.66%, heating value (combined mass) 8044 cal.; semi-coke 34.5, tar on the combustible mass 52.2, H_2O of decompn. 3.5 and gas and losses 25.3%. The Moscow coal analyzed: H_2O 7.80, ash 33.74, total S 3.76, C 63.72, H 5.21 and volatile matter 65.67%. Carbonizing (*a*) at 410–460° during 90–250 min. and 0–50 atm. yielded: residual coal 49.0–60.3, distillate 9.7–20.0 and H_2O 6.5–8.2%; the gas gasoline amounted to 1.7% gas and losses 15.8–24.9%. The distillate yielded: 7.7–10.1% gasoline (on coal) of 0.763–0.774 sp. gr. Hydrogenation of the coal at 384–450°, for 30–240 min., 118–223 atm. pressure in the presence or absence of catalysts, such as 10% NiO or 0.5–5.0% NH_4 molybdate, yielded: gas gasoline 0.7–3.5, residue 25.5–70.6 (contg. coal 31.0–37.5 and oil 0.9–39.1%), total yield of liquid products 31.9–40.0 and gas and losses 13.5–43.7%. The hydrogenation of (*b*) with the above catalyst at 380–480°, other conditions remaining the same, yielded 6% of gasoline after 2 recyclings; and the 4th recycling yielded another 6–7%. The total yield of liquid products amounted to 12–14% and 3.8% more oil was extd. with C_6H_6. Hydrogenation of the supromixite treated with HF in the presence of NH_4 molybdate catalyst yielded: distillate 46, H_2O 2.3, coal 31.2 and losses 27.4%. The liquid fraction contained: gasoline 12.4–34.8, kerosene 21.7–47.9 and residue 16.1–49.5%.

A. A. Boehtlingk



Characteristics of gasolines obtained in the destructive hydrogenation of sapropelitic tar. I. P. Rapoport and N. V. Mikhailova. Akad. Nauk Belorussk. S.S.R., Trudy Inst. Torfu 1, 5-16(1940). In the destructive hydrogenation of sapropelitic tars, the gasolines produced contained aromatic hydrocarbons such as C_6H_6, $PhMe$, etc., as well as p-$C_6H_4Me_2$ and apparently $PhEt$. Among the unsatd. compds., those with C_5 to C_{12} with a double bond at the 2nd C atom were identified. The presence of 5- and 6-membered naphthenes and of hydrocarbons with a tertiary C atom was established. A. A. Bochkarev



Ca

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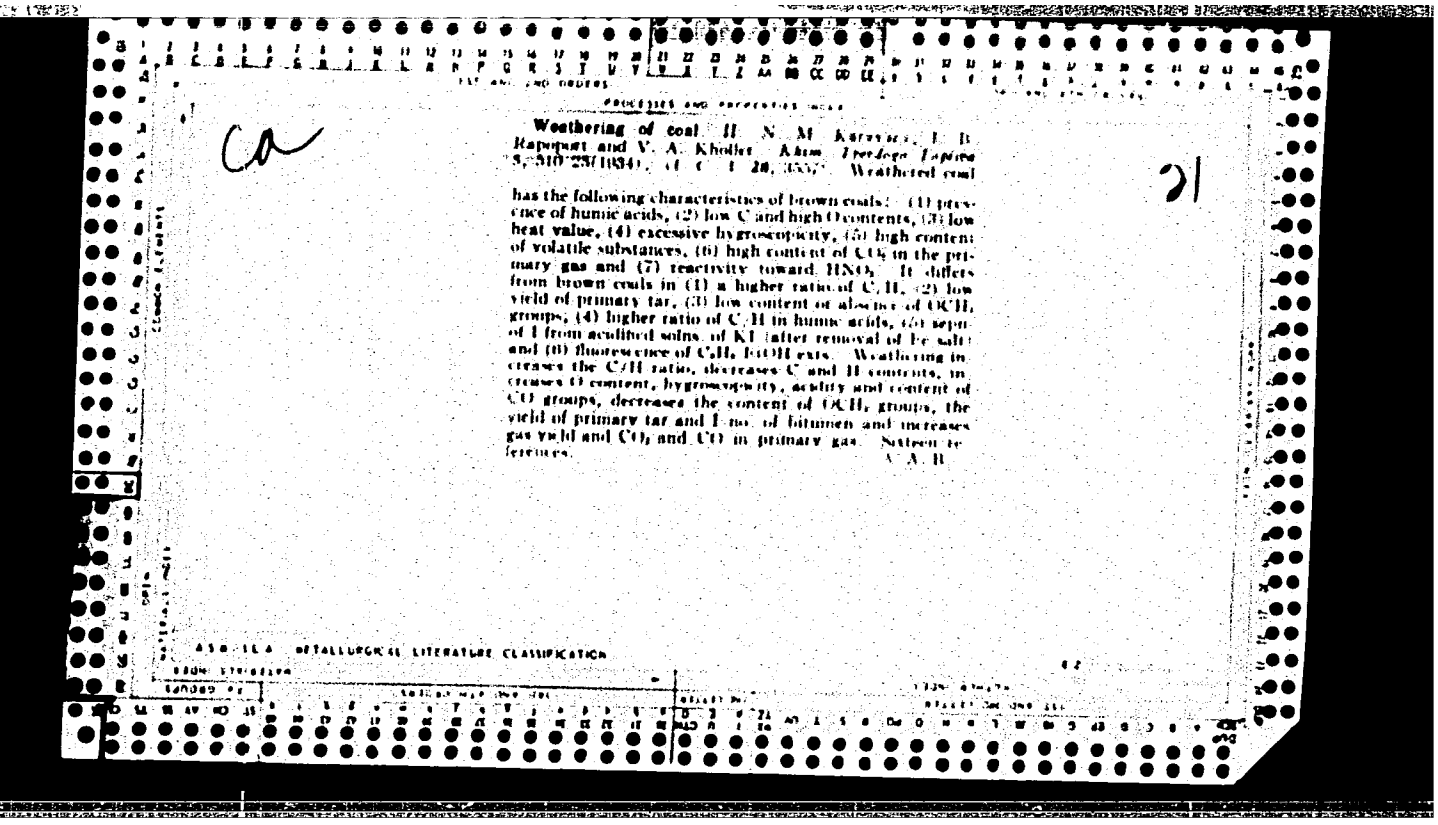
The bases of primary tar from Zhurinsk coals. L. B. Margaret and Z. H. Kondapov. *Khim. Tverdogo Topliva* 5 [1934]; cf. *C. A.* 28, 6280⁹.—Zhurinsk coal yielded in a gas producer 12–15% of a tar of d_{15}^{15} 1.0179. The kerosene admixed with some of the heavier fractions obtained on redistn. of this tar was treated twice with 15% H_2SO_4, the phenols were removed and the product was treated twice with 20% H_2SO_4. Free bases were suspd. from the two last exts., coagd., steam-distd. and treated with Et_2O. The product was decompd. with 30% caustic, the suspd. bases distd. with superheated steam, the residue treated with Et_2O and the latter distd. off. The bases were again treated with H_2SO_4 and the soln. treated again with superheated steam and Et_2O. The salts obtained were decompd. in a stream of NH_3. The upper layer of bases was suspd., and the bases in the soln. were extd. with Et_2O and added to the main portion. The light bases were suspd. from the heavy by steam distn. Thus were obtained light bases of d_4^{15} 0.9972 and heavy bases of d_4^{15} 1.0020. The mol. wts. of various fractions distd. with N HCl were: fraction b. 180–190° 112.87, 190–200° 114.62, 200–210° 116.91, 210–220° 118.55 and 220–230° 125.99. Narrow cuts (after vacuum redistn.) were converted into picrates and their mol. wts. were detd. by titration with 0.1 *N* KOH. The picrate fraction b. 230–237° corresponded to methylquinoline. The base fraction b. at 200–220° was hydrogenated with 5% of Mo oxide, in an Ipatieff bomb at 450°. In the product hydrogenated tertiary bases were absent but $PhNMe_2$, $PhNHMe$ and primary amines were present.

A. A. Bashtinsk



Ca

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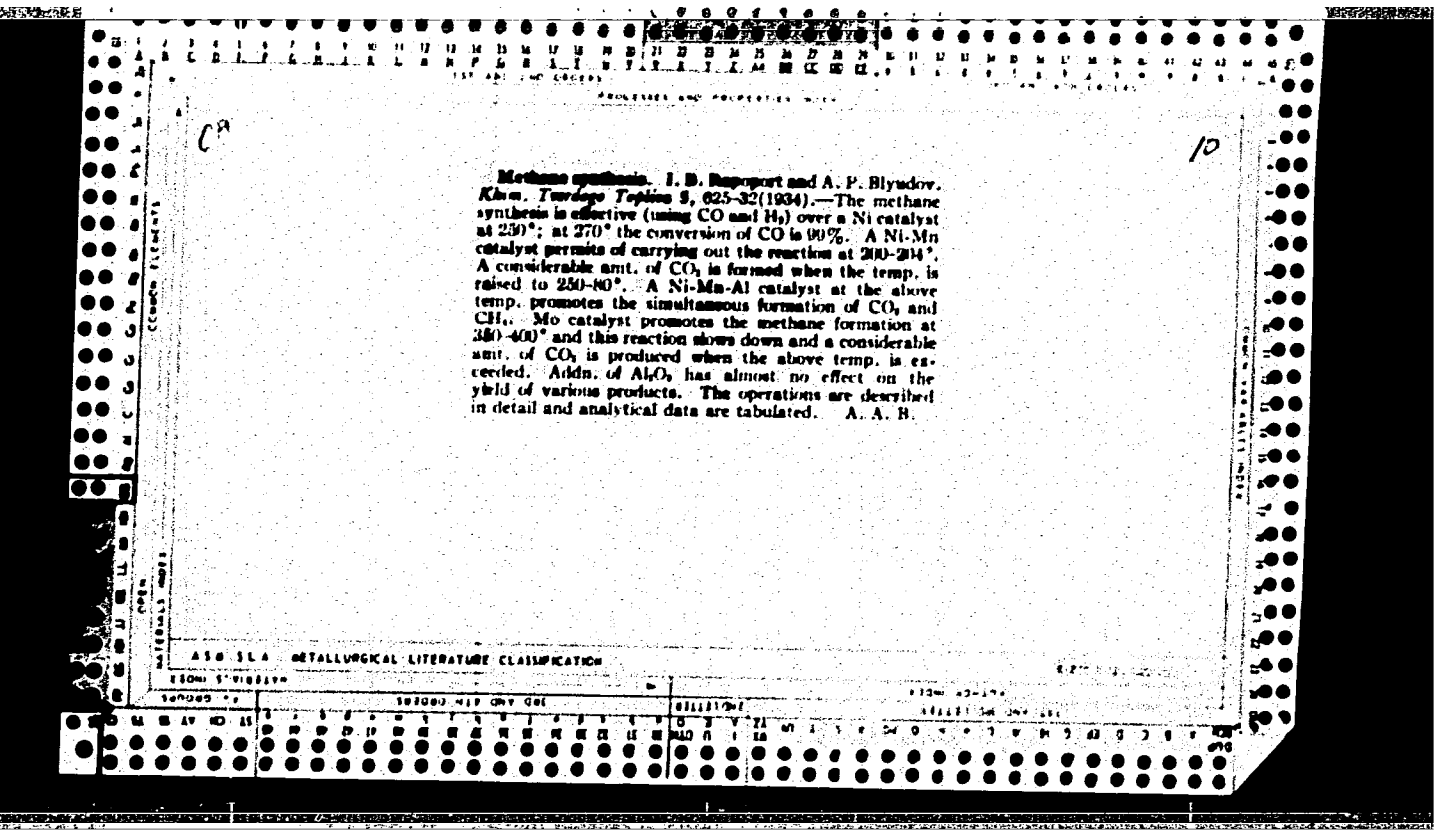
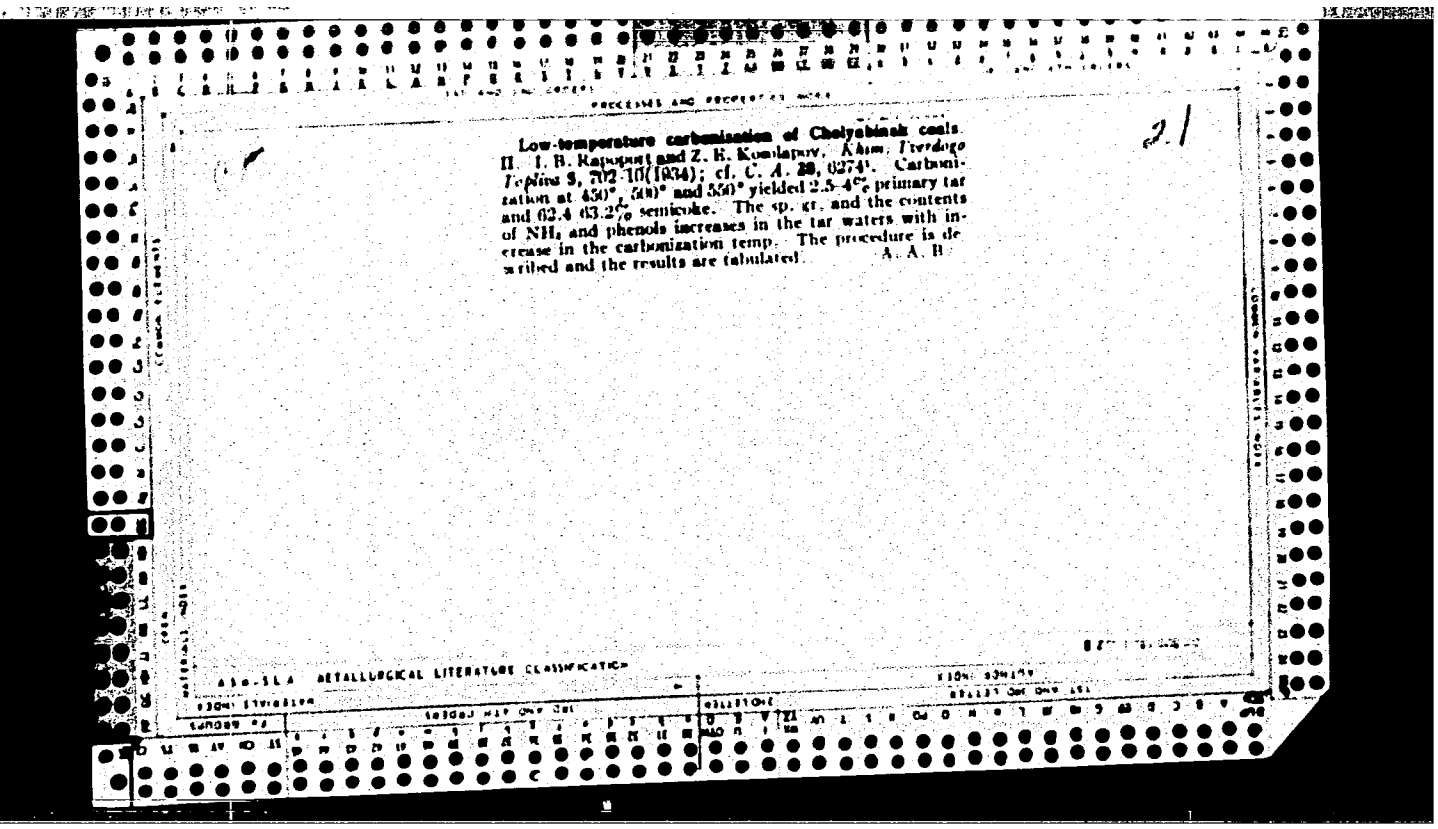
Weathering of coal II. N. M. Karyakin, I. B. Rapoport and V. A. Khodler. *Khim. Tverdogo Topliva* 5, 310-25(1934). (Russ.) Weathered coal has the following characteristics of brown coals: (1) presence of humic acids, (2) low C and high O contents, (3) low heat value, (4) excessive hygroscopicity, (5) high content of volatile substances, (6) high content of CO_2 in the primary gas and (7) reactivity toward HNO_3. It differs from brown coals in (1) a higher ratio of C/H, (2) low yield of primary tar, (3) low content or absence of OCH$_3$ groups, (4) higher ratio of C/H in humic acids, (5) separation of I from acidified solns. of KI (after removal of Fe salts) and (6) fluorescence of C_6H_5OH extn. Weathering increases the C/H ratio, decreases C and H contents, increases O content, hygroscopicity, acidity and content of CO groups, decreases the content of OCH$_3$ groups, the yield of primary tar and [illegible] of bitumen and increases gas yield and CO_2 and CO in primary gas. Sixteen references. A. A. B.

A 5 0 - 11 A METALLURGICAL LITERATURE CLASSIFICATION



Methane synthesis. I. B. Rapoport and A. P. Blyudov. *Khim. Tverdogo Topliva* 5, 625-32(1934).—The methane synthesis is effective (using CO and H_2) over a Ni catalyst at 250°; at 270° the conversion of CO is 90%. A Ni-Mn catalyst permits of carrying out the reaction at 200-204°. A considerable amt. of CO_2 is formed when the temp. is raised to 250-80°. A Ni-Mn-Al catalyst at the above temp. promotes the simultaneous formation of CO_2 and CH_4. Mo catalyst promotes the methane formation at 380-400° and this reaction slows down and a considerable amt. of CO_2 is produced when the above temp. is exceeded. Addn. of Al_2O_3 has almost no effect on the yield of various products. The operations are described in detail and analytical data are tabulated. A. A. B.



Low-temperature carbonization of Cheiyabinsk coals. II. I. B. Rapoport and Z. E. Kumskaya. *Khim. Tverdogo Topliva* 5, 707-10(1934); cf. *C. A.* 28, 6274. Carbonization at 450°, 500° and 550° yielded 2.5-4% primary tar and 62.4-63.2% semicoke. The sp. gr. and the contents of NH_3 and phenols increases in the tar waters with increase in the carbonization temp. The procedure is described and the results are tabulated. A. A. B.

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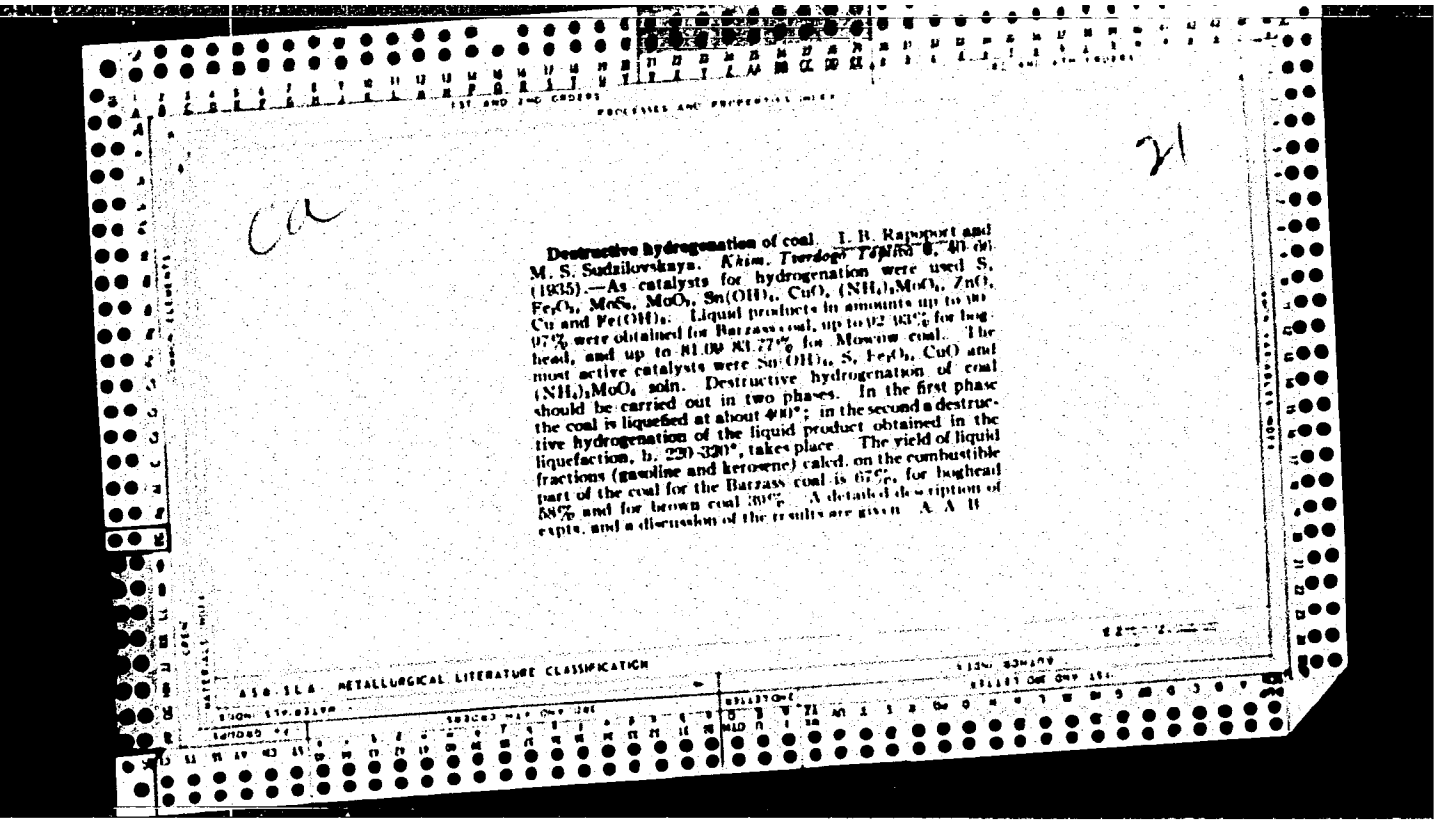
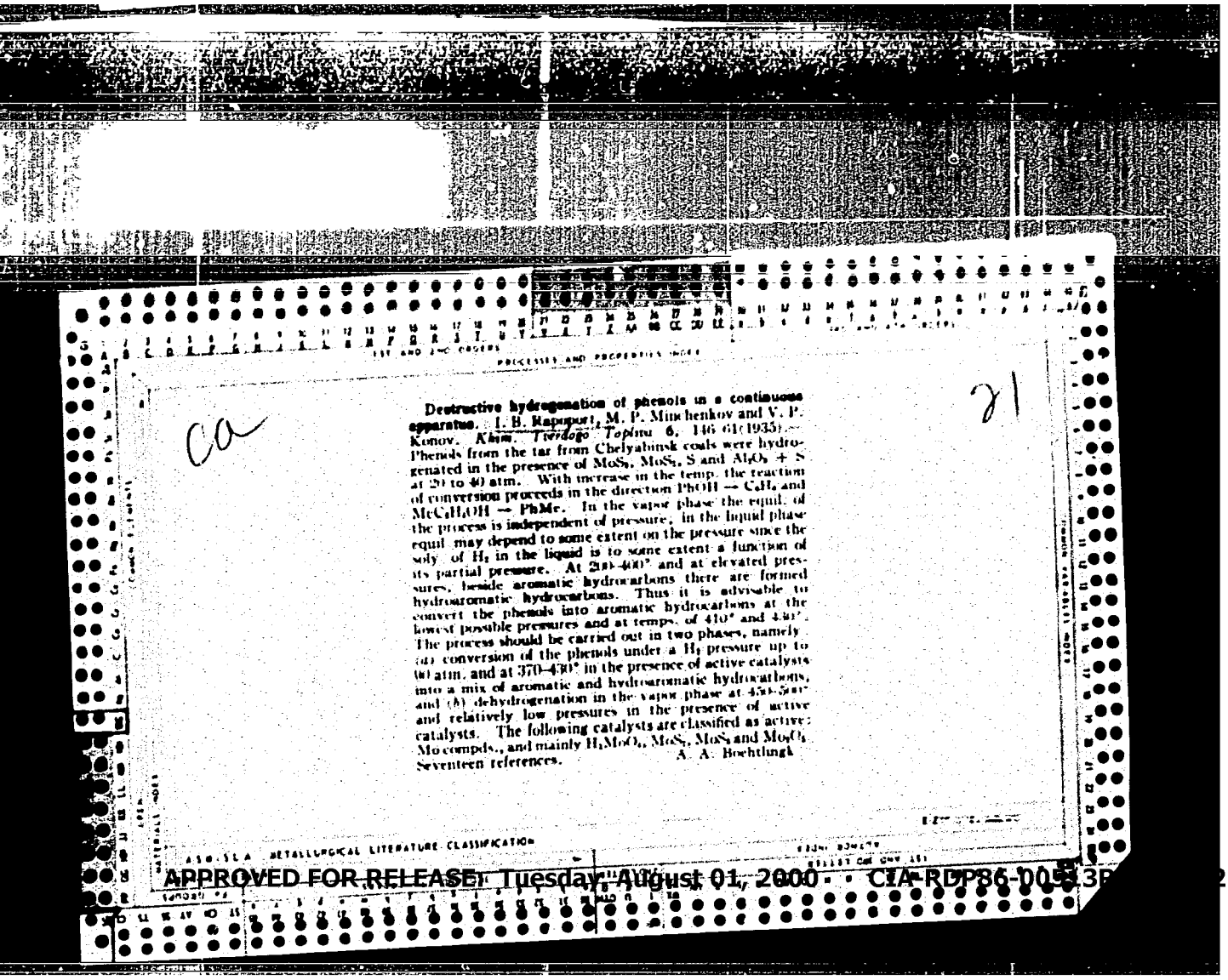
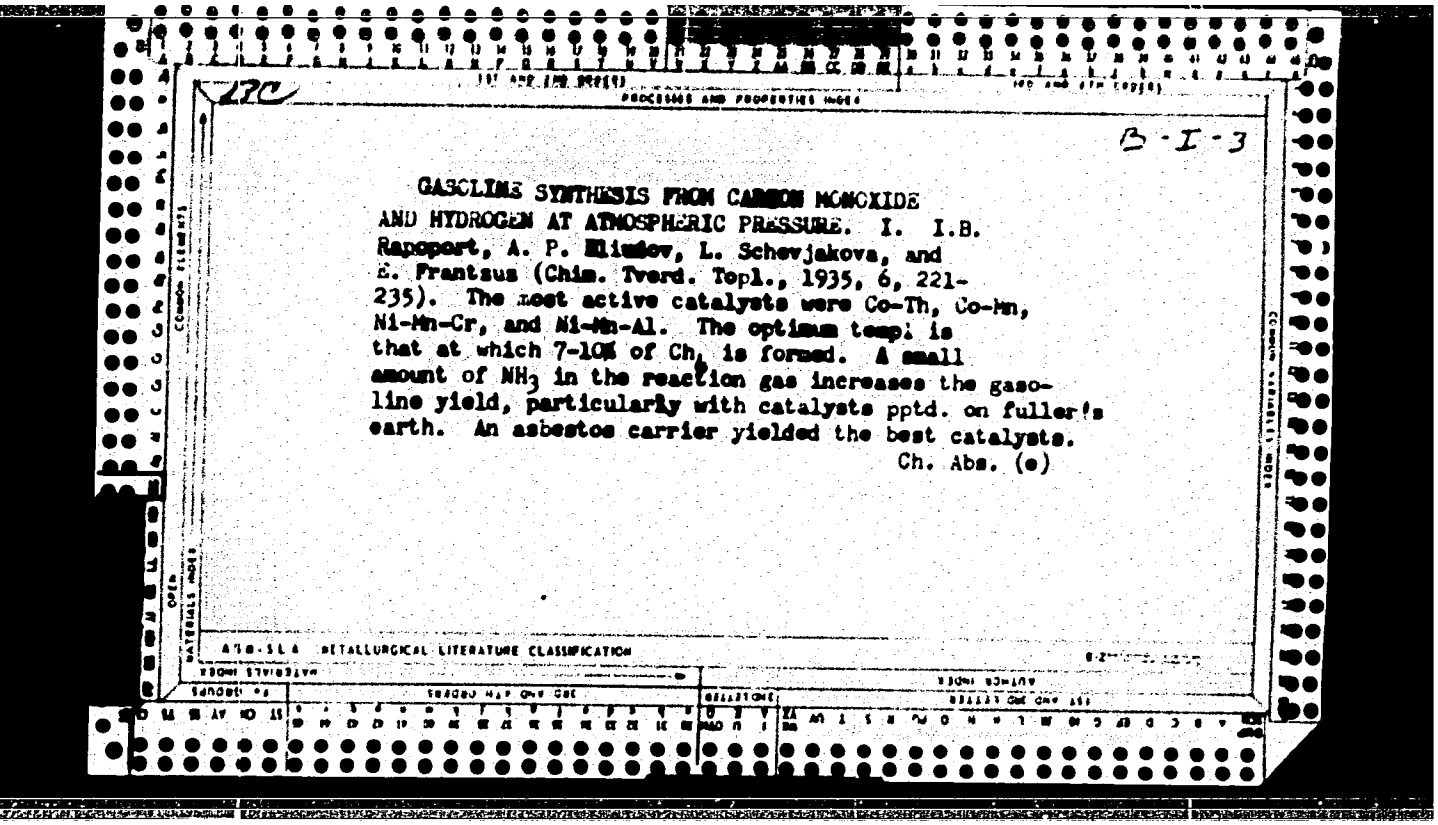


Destructive hydrogenation of coal. I. B. Rapoport and M. S. Sudnikovskaya. *Khim. Tverdogo Topliva* 6, 40-61 (1935).—As catalysts for hydrogenation were used S, Fe_2O_3, MoS_2, MoO_3, $Sn(OH)_4$, CuO, $(NH_4)_2MoO_4$, ZnO, Cu and $Fe(OH)_3$. Liquid products in amounts up to 90-97% were obtained for Barzass coal, up to 92-93% for bughead, and up to 81.09-83.77% for Moscow coal. The most active catalysts were $Sn(OH)_4$, S, Fe_2O_3, CuO and $(NH_4)_2MoO_4$ soln. Destructive hydrogenation of coal should be carried out in two phases. In the first phase the coal is liquefied at about 400°; in the second a destructive hydrogenation of the liquid product obtained in the liquefaction, b. 250-320°, takes place. The yield of liquid fractions (gasoline and kerosene) calcd. on the combustible part of the coal for the Barzass coal is 67%, for bughead 58% and for brown coal 30%. A detailed description of expts. and a discussion of the results are given. A. A. B.



Destructive hydrogenation of phenols in a continuous apparatus. I. B. Rapoport, M. P. Minchenkov and V. P. Konov. *Khim. Tverdogo Topliva* 6, 146-61(1935).—Phenols from the tar from Chelyabinsk coals were hydrogenated in the presence of MoS_3, MoS_2, S and Al_2O_3 + S at 20 to 40 atm. With increase in the temp. the reaction of conversion proceeds in the direction $PhOH \rightarrow C_6H_6$ and $MeC_6H_4OH \rightarrow PhMe$. In the vapor phase the equil. of the process is independent of pressure, in the liquid phase equil. may depend to some extent on the pressure since the soly. of H_2 in the liquid is to some extent a function of its partial pressure. At 280-400° and at elevated pressures, beside aromatic hydrocarbons there are formed hydroaromatic hydrocarbons. Thus it is advisable to convert the phenols into aromatic hydrocarbons at the lowest possible pressures and at temps. of 410° and 440°. The process should be carried out in two phases, namely (a) conversion of the phenols under a H_2 pressure up to 60 atm. and at 370-430° in the presence of active catalysts into a mix of aromatic and hydroaromatic hydrocarbons, and (b) dehydrogenation in the vapor phase at 450-500° and relatively low pressures in the presence of active catalysts. The following catalysts are classified as active: Mo compds., and mainly H_2MoO_4, MoS_2, MoS_3 and MoO_3. Seventeen references.

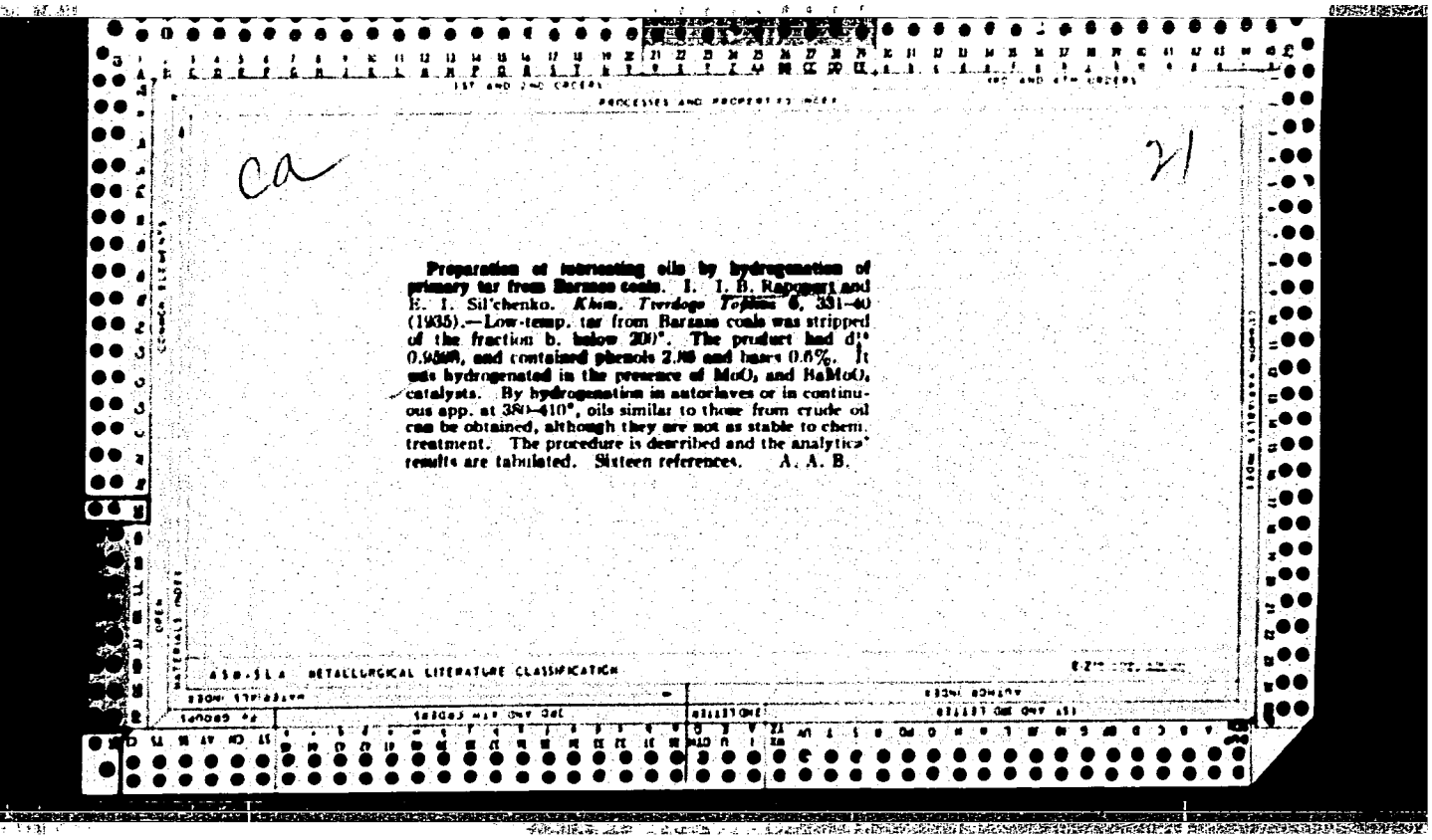
A. A. Brechtlingk



B-I-3

GASOLINE SYNTHESIS FROM CARBON MONOXIDE AND HYDROGEN AT ATMOSPHERIC PRESSURE. I. I.B. Rapoport, A. P. Miliudov, L. Schevjakova, and E. Frantsus (Chim. Tverd. Topl., 1935, 6, 221-235). The most active catalysts were Co-Th, Co-Mn, Ni-Mn-Cr, and Ni-Mn-Al. The optimum temp. is that at which 7-10% of Ch_4 is formed. A small amount of NH_3 in the reaction gas increases the gasoline yield, particularly with catalysts pptd. on fuller's earth. An asbestos carrier yielded the best catalysts.

Ch. Abs. (e)



Ca

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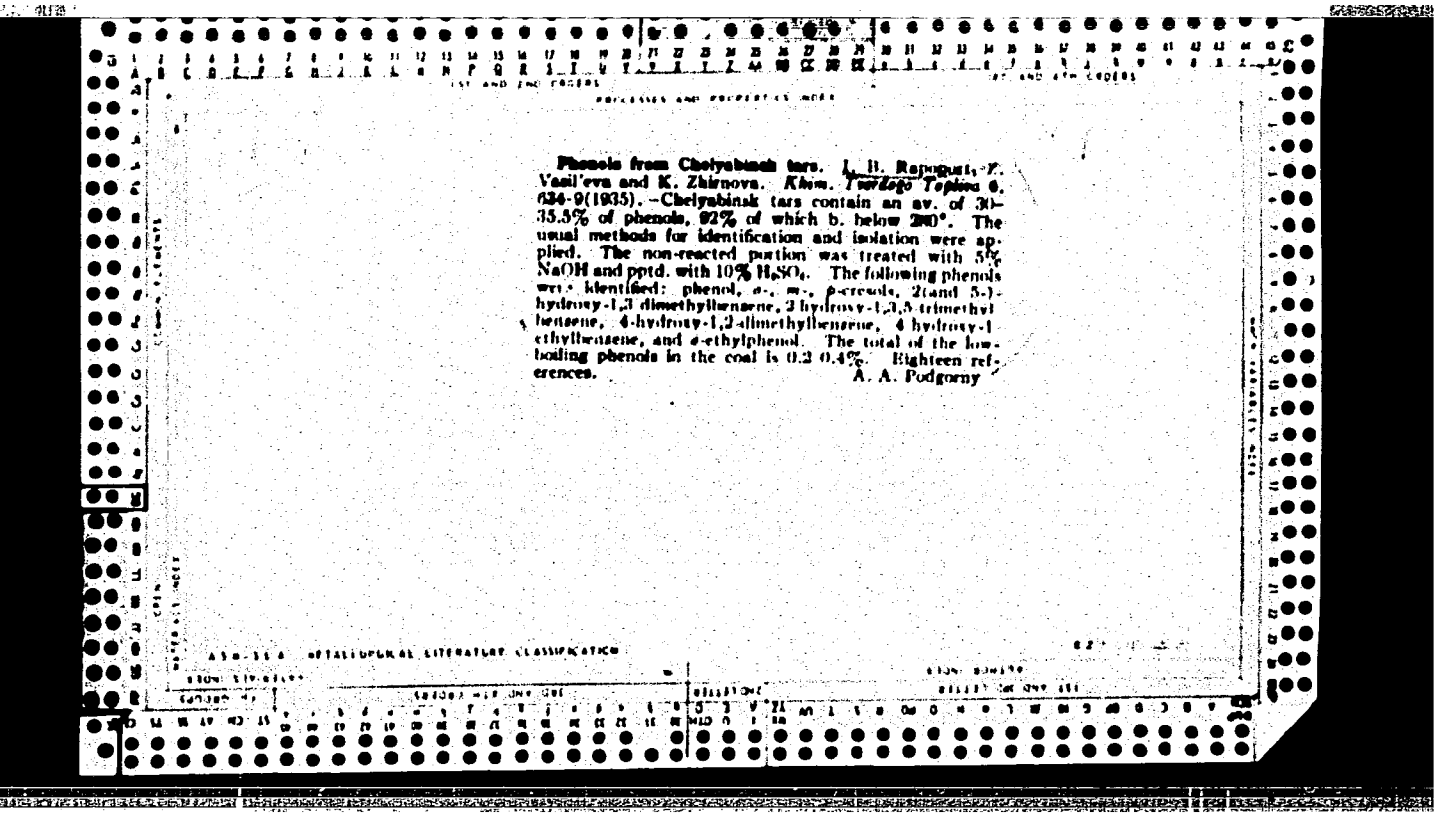
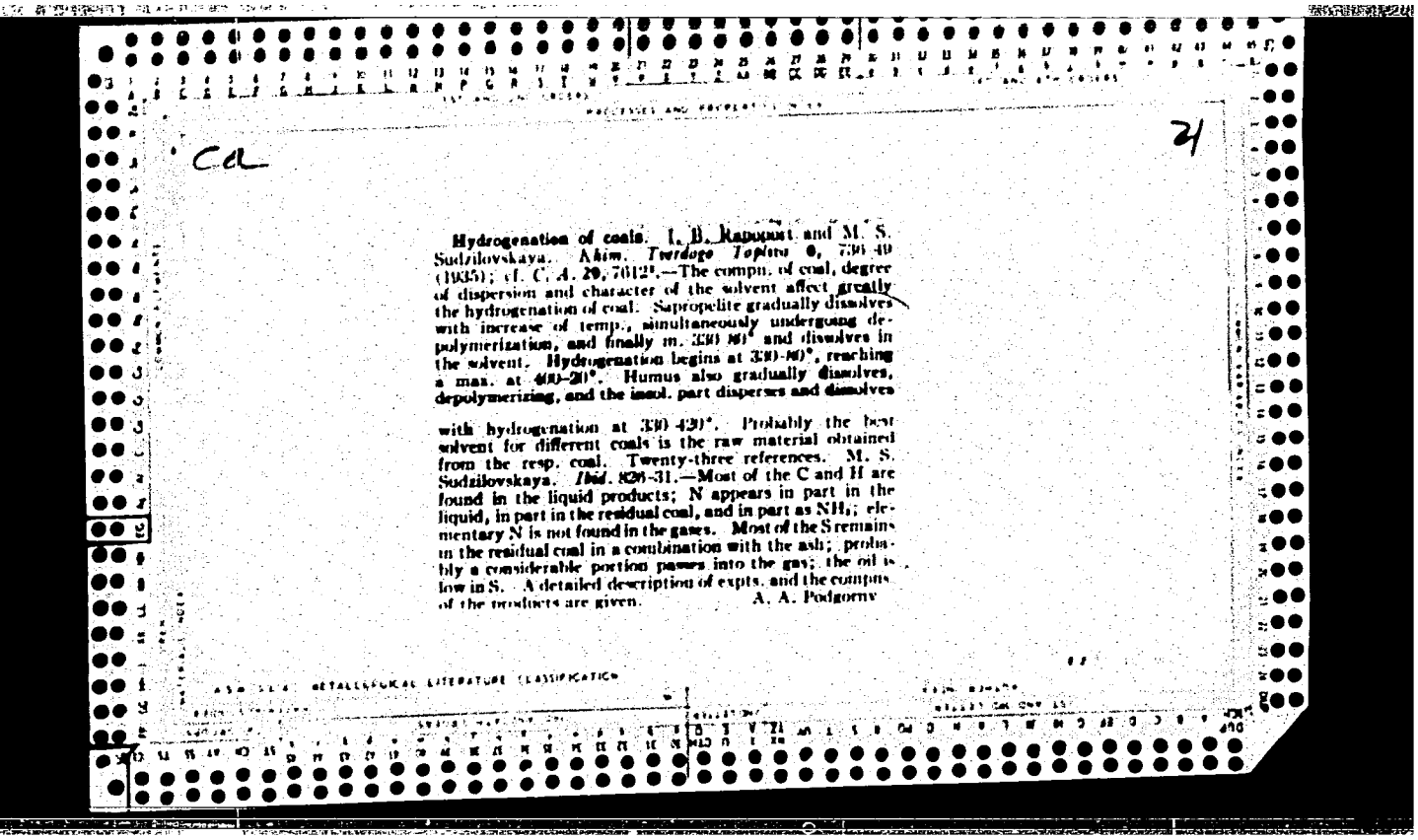
Preparation of intermediate oils by hydrogenation of primary tar from Barzass coals. I. I. B. Rapoport and E. I. Sil'chenko. *Khim. Tverdogo Topliva* 6, 331–40 (1935).—Low-temp. tar from Barzass coals was stripped of the fraction b. below 200°. The product had d_4^{20} 0.9569, and contained phenols 2.0% and bases 0.6%. It was hydrogenated in the presence of MoO$_3$ and NaMoO$_4$ catalysts. By hydrogenation in autoclaves or in continuous app. at 380–410°, oils similar to those from crude oil can be obtained, although they are not as stable to chem. treatment. The procedure is described and the analytical results are tabulated. Sixteen references. A. A. B.

ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION



Phenols from Cheiyabinsk tars. I. N. Rapoport, V. Vasil'eva and K. Zhirnova. *Khim. Tverdogo Topliva* 6, 634-9(1935).—Cheiyabinsk tars contain an av. of 30–35.5% of phenols, 92% of which b. below 280°. The usual methods for identification and isolation were applied. The non-reacted portion was treated with 5% NaOH and pptd. with 10% H_2SO_4. The following phenols were identified: phenol, o-, m-, p-cresols, 2(and 5-)-hydroxy-1,3 dimethylbenzene, 2 hydroxy-1,3,5-trimethylbenzene, 4-hydroxy-1,2-dimethylbenzene, 4 hydroxy-1-ethylbenzene, and o-ethylphenol. The total of the low-boiling phenols in the coal is 0.2 0.4%. Eighteen references.

A. A. Podgorny



Cal

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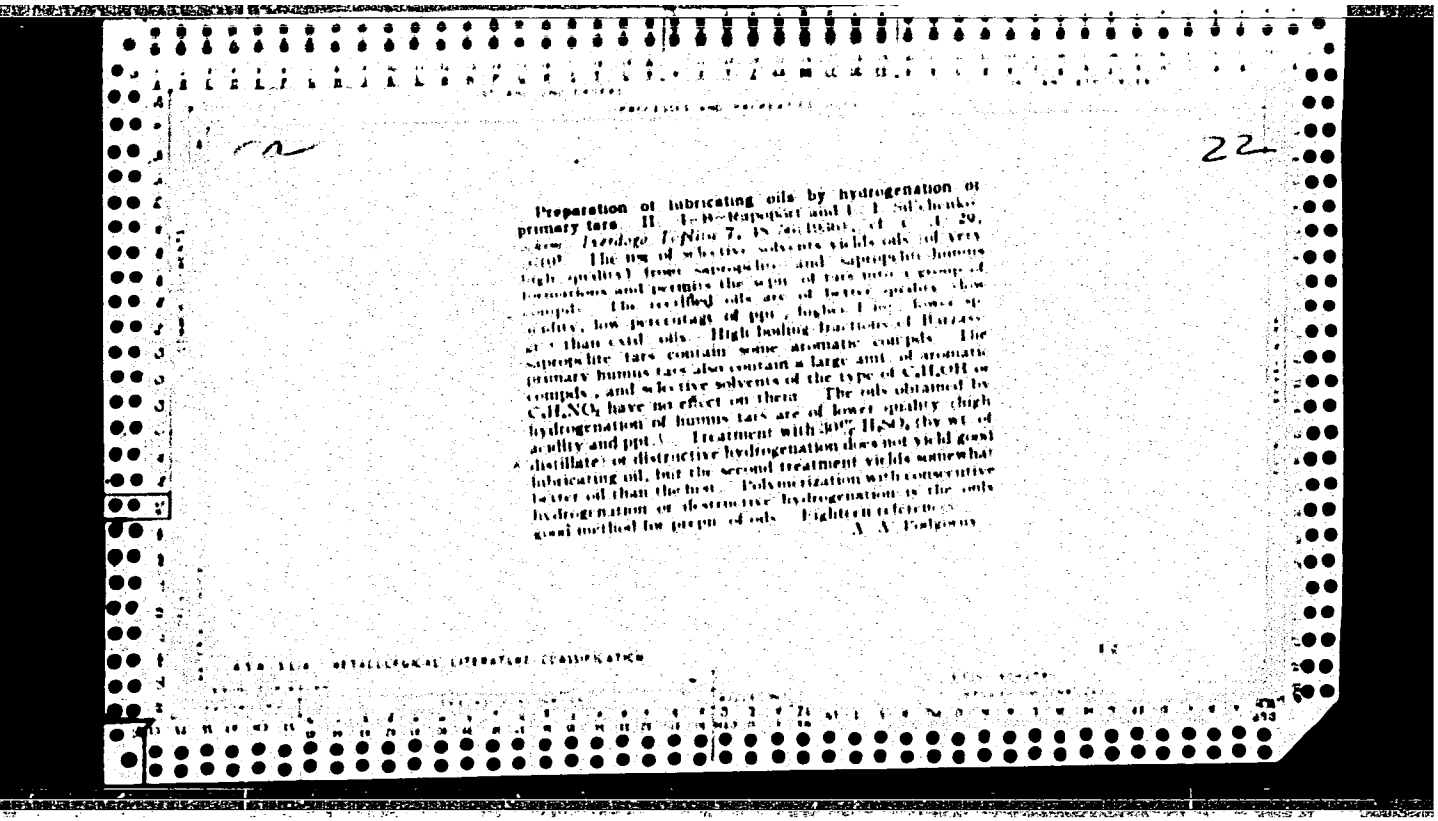
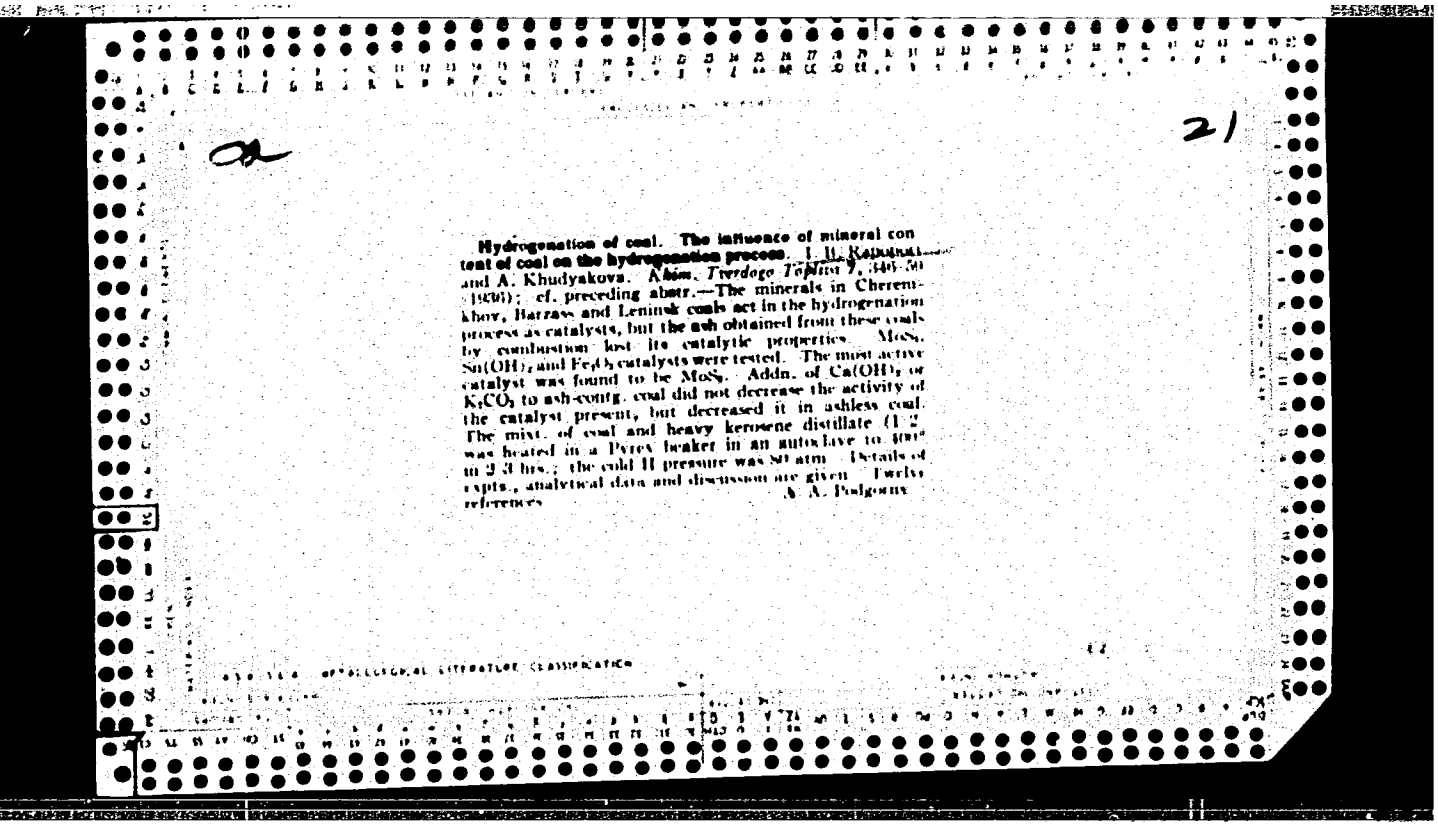
Hydrogenation of coals. I. B. Rapoport and M. S. Sudzilovskaya. *Khim. Tverdogo Topliva* **6**, 736-49 (1935); cf. *C. A.* **29**, 7012⁸.—The compn. of coal, degree of dispersion and character of the solvent affect greatly the hydrogenation of coal. Sapropelite gradually dissolves with increase of temp., simultaneously undergoing depolymerization, and finally in. 330 at 380° and dissolves in the solvent. Hydrogenation begins at 330-80°, reaching a max. at 400-20°. Humus also gradually dissolves, depolymerizing, and the insol. part disperses and dissolves with hydrogenation at 330-420°. Probably the best solvent for different coals is the raw material obtained from the resp. coal. Twenty-three references. M. S. Sudzilovskaya. *Ibid.* 826-31.—Most of the C and H are found in the liquid products; N appears in part in the liquid, in part in the residual coal, and in part as NH_3; elementary N is not found in the gases. Most of the S remains in the residual coal in a combination with the ash; probably a considerable portion passes into the gas; the oil is low in S. A detailed description of expts. and the compns. of the products are given. A. A. Podgorny

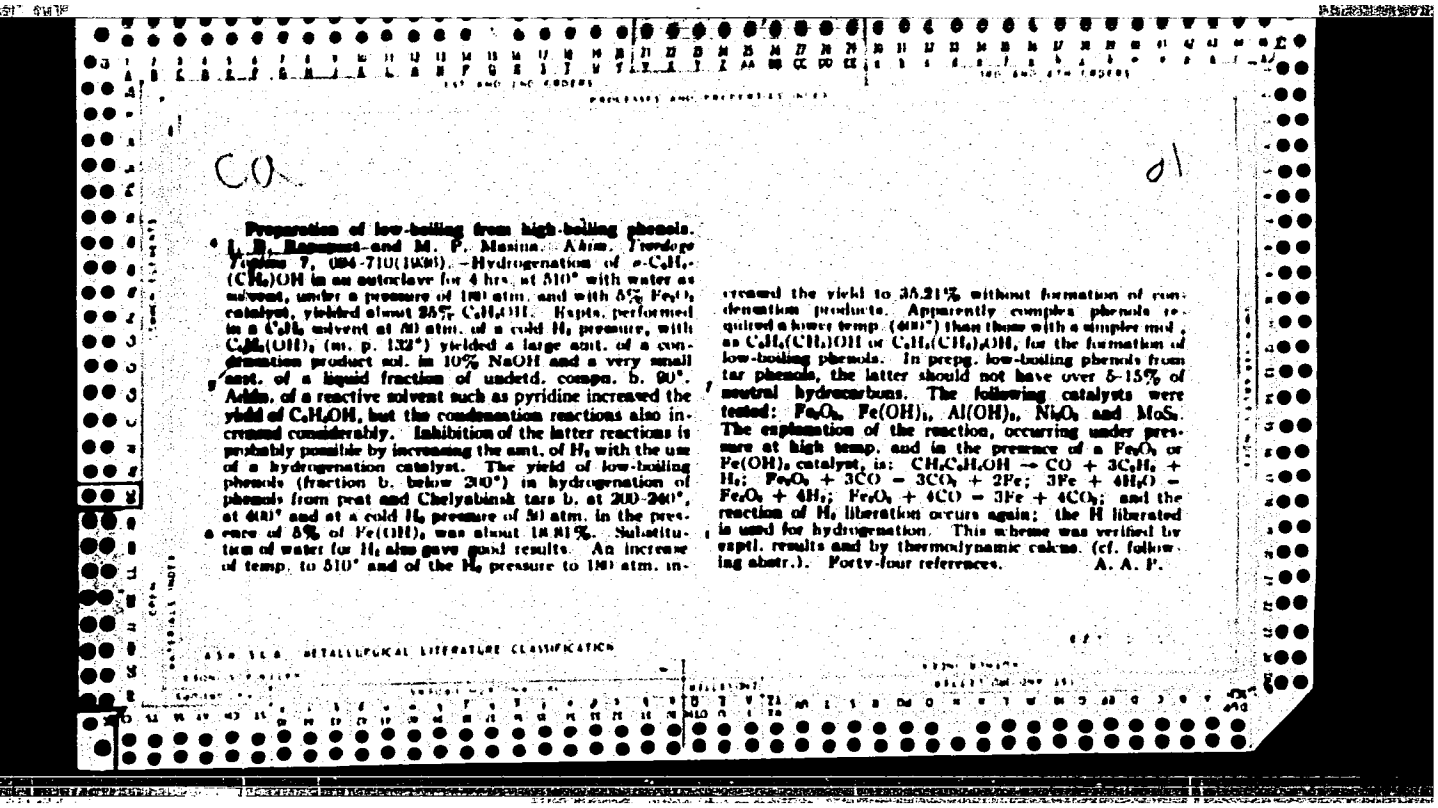
METALLURGICAL LITERATURE CLASSIFICATION



Preparation of lubricating oils by hydrogenation of primary tars. H. [illegible] and I. I. Sil'chenkov. *Trudy Inst. Goryuchego Iskopaemykh, Akad. Nauk S.S.S.R.* 7, 19 (1938); cf. C. A. 32, 29. The use of selective solvents yields oils of very high quality from sapropelite and sapropelitic humus hydrocarbons and permits the use of tars from a greater number of coals. The rectified oils are of better quality, low percentage of ppt., higher flash point, lower specific gravity than cracked oils. High boiling fractions of Harass sapropelite tars contain some aromatic compds. The primary humus tars also contain a large amt. of aromatic compds., and selective solvents of the type of C_6H_5OH or $C_6H_5NO_2$ have no effect on them. The oils obtained by hydrogenation of humus tars are of lower quality (high acidity and ppt.). Treatment with 30% H_2SO_4 (by wt. of distillate) or destructive hydrogenation does not yield good lubricating oil, but the second treatment yields somewhat better oil than the first. Polymerization with conservative hydrogenation or destructive hydrogenation is the only good method for prepn. of oils. Eighteen references. A. V. Palgunov



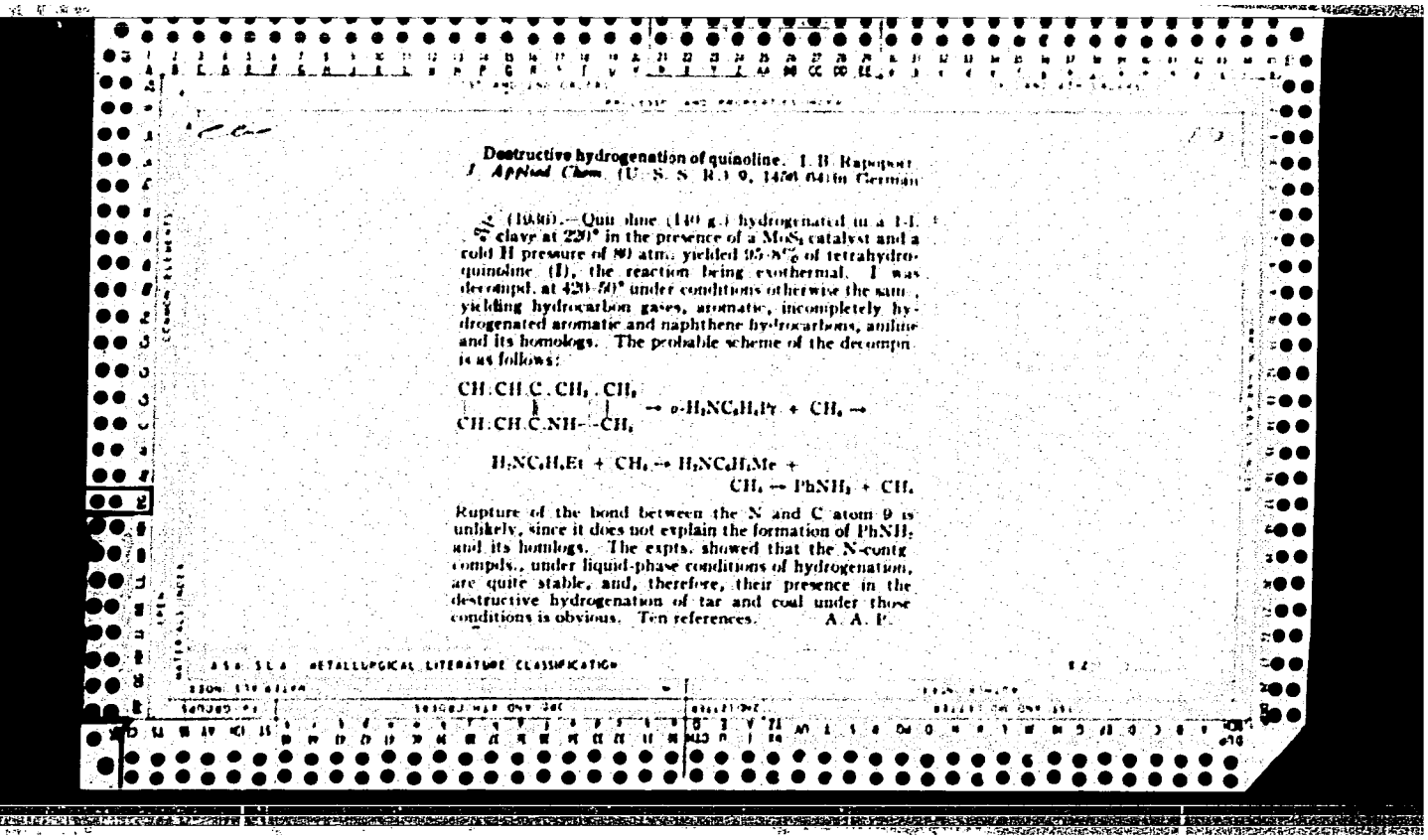
Hydrogenation of coal. The influence of mineral content of coal on the hydrogenation process. I. H. Kapustina and A. Khudyakova. *Khim. Tverdogo Topliva* 7, 346-50 (1936); cf. preceding abstr.—The minerals in Cheremkhov, Barzass and Leninsk coals act in the hydrogenation process as catalysts, but the ash obtained from these coals by combustion lost its catalytic properties. MoS_2, $Sn(OH)_2$ and Fe_2O_3 catalysts were tested. The most active catalyst was found to be MoS_2. Addn. of $Ca(OH)_2$ or K_2CO_3 to ash-contg. coal did not decrease the activity of the catalyst present, but decreased it in ashless coal. The mixt. of coal and heavy kerosene distillate (1:2) was heated in a Pyrex beaker in an autoclave to 400° in 2.3 hrs.; the cold H pressure was 50 atm. Details of expts., analytical data and discussion are given. Twelve references. A. A. Bulgakov



CO

01

Preparation of low-boiling from high-boiling phenols. L. B. Rasnopust and M. P. Masina. *Khim. Tverdogo Topliva* 7, 694-710(1936). —Hydrogenation of *o*-$C_6H_4(CH_3)OH$ in an autoclave for 4 hrs. at 510° with water as solvent, under a pressure of 180 atm. and with 5% $Fe(OH)_3$ catalyst, yielded about 35% C_6H_5OH. Expts. performed in a C_6H_6 solvent at 50 atm. of a cold H_2 pressure, with $C_6H_4(OH)_2$ (m. p. 132°) yielded a large amt. of a condensation product sol. in 10% NaOH and a very small amt. of a liquid fraction of undetd. compn. b. 90°. Addn. of a reactive solvent such as pyridine increased the yield of C_6H_5OH, but the condensation reactions also increased considerably. Inhibition of the latter reactions is probably possible by increasing the amt. of H_2 with the use of a hydrogenation catalyst. The yield of low-boiling phenols (fraction b. below 230°) in hydrogenation of phenols from peat and Chelyabinsk tars b. at 200-240°, at 440° and at a cold H_2 pressure of 50 atm. in the presence of 5% of $Fe(OH)_3$ was about 18.81%. Substitution of water for H_2 also gave good results. An increase of temp. to 510° and of the H_2 pressure to 180 atm. increased the yield to 35.21% without formation of condensation products. Apparently complex phenols required a higher temp. (440°) than those with a simpler mol., as $C_6H_4(CH_3)OH$ or $C_6H_4(CH_3)_2OH$, for the formation of low-boiling phenols. In prepg. low-boiling phenols from tar phenols, the latter should not have over 8-15% of neutral hydrocarbons. The following catalysts were tested: Fe_2O_3, $Fe(OH)_3$, $Al(OH)_3$, Ni_2O_3 and MoS_2. The explanation of the reaction, occurring under pressure at high temp. and in the presence of a Fe_2O_3 or $Fe(OH)_3$ catalyst, is: $CH_3C_6H_4OH \rightarrow CO + 3C_2H_6 + H_2$; $Fe_2O_3 + 3CO = 3CO_2 + 2Fe$; $3Fe + 4H_2O = Fe_3O_4 + 4H_2$; $Fe_3O_4 + 4CO = 3Fe + 4CO_2$; and the reaction of H_2 liberation occurs again; the H liberated is used for hydrogenation. This scheme was verified by exptl. results and by thermodynamic calcns. (cf. following abstr.). Forty-four references. A. A. P.



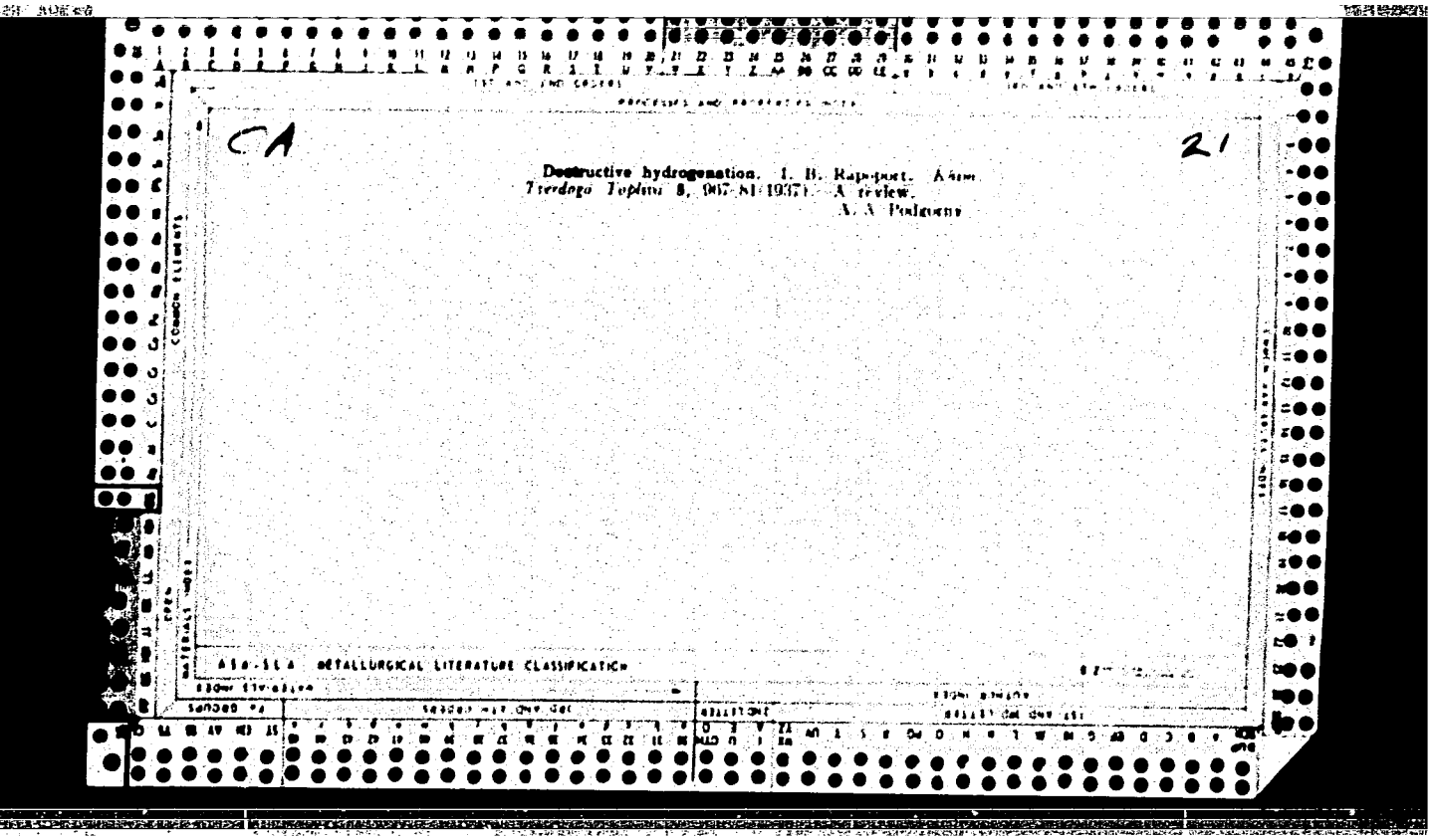
Destructive hydrogenation of quinoline. I. B. Rapoport. *J. Applied Chem.* (U. S. S. R.) 9, 1450-64 (in German)

(1936).—Quinoline (140 g.) hydrogenated in a 1-l. steel clave at 220° in the presence of a MoS_2 catalyst and a cold H pressure of 80 atm. yielded 95-98% of tetrahydroquinoline (I), the reaction being exothermal. I was decompd. at 420-50° under conditions otherwise the same, yielding hydrocarbon gases, aromatic, incompletely hydrogenated aromatic and naphthene hydrocarbons, aniline and its homologs. The probable scheme of the decompn. is as follows:

$$\begin{matrix} CH{:}CH.C(CH_3){:}CH_2 \\ | \qquad\qquad | \\ CH{:}CH.C(NH{-}){-}CH_2 \end{matrix} \rightarrow o\text{-}H_2NC_6H_4Pr + CH_4 \rightarrow$$

$$H_2NC_6H_4Et + CH_4 \rightarrow H_2NC_6H_4Me + CH_4 \rightarrow PhNH_2 + CH_4$$

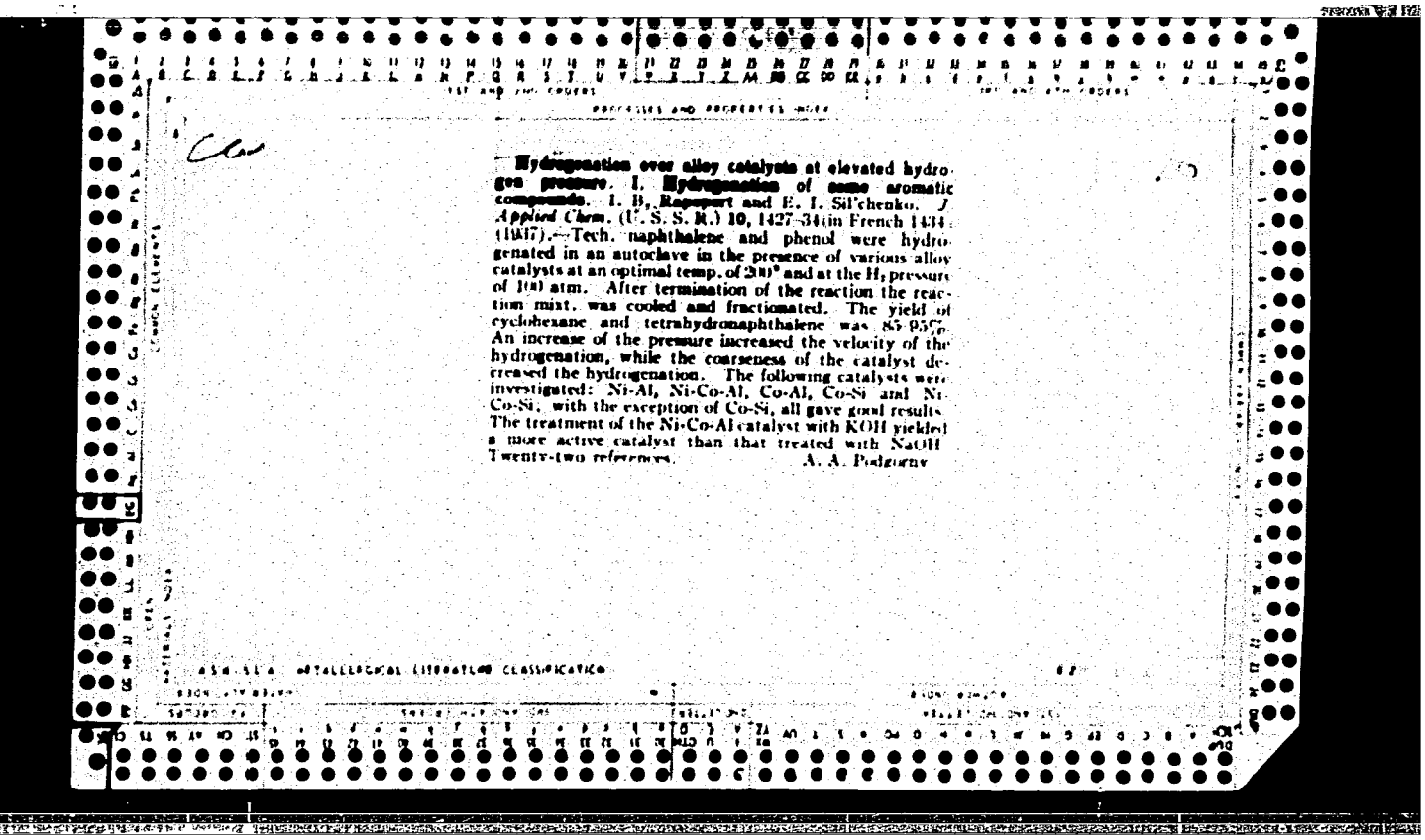
Rupture of the bond between the N and C atom 9 is unlikely, since it does not explain the formation of $PhNH_2$ and its homologs. The expts. showed that the N-contg. compds., under liquid-phase conditions of hydrogenation, are quite stable, and, therefore, their presence in the destructive hydrogenation of tar and coal under those conditions is obvious. Ten references. A. A. P.



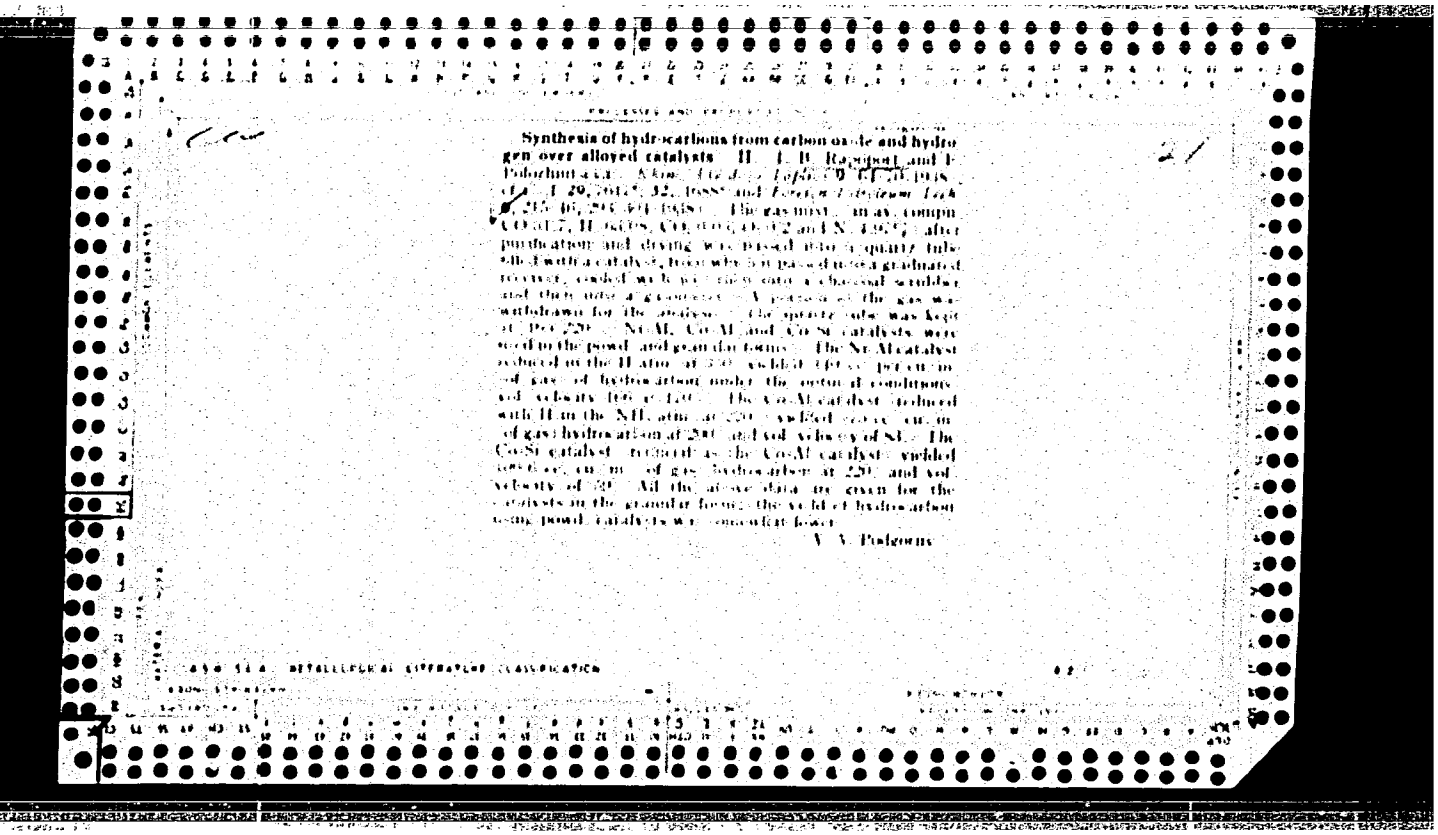
CA

21

Destructive hydrogenation. I. B. Rapoport. Azim. Tverdogo Topliva 8, 907-81 (1937). A review.
A. A. Podgorny

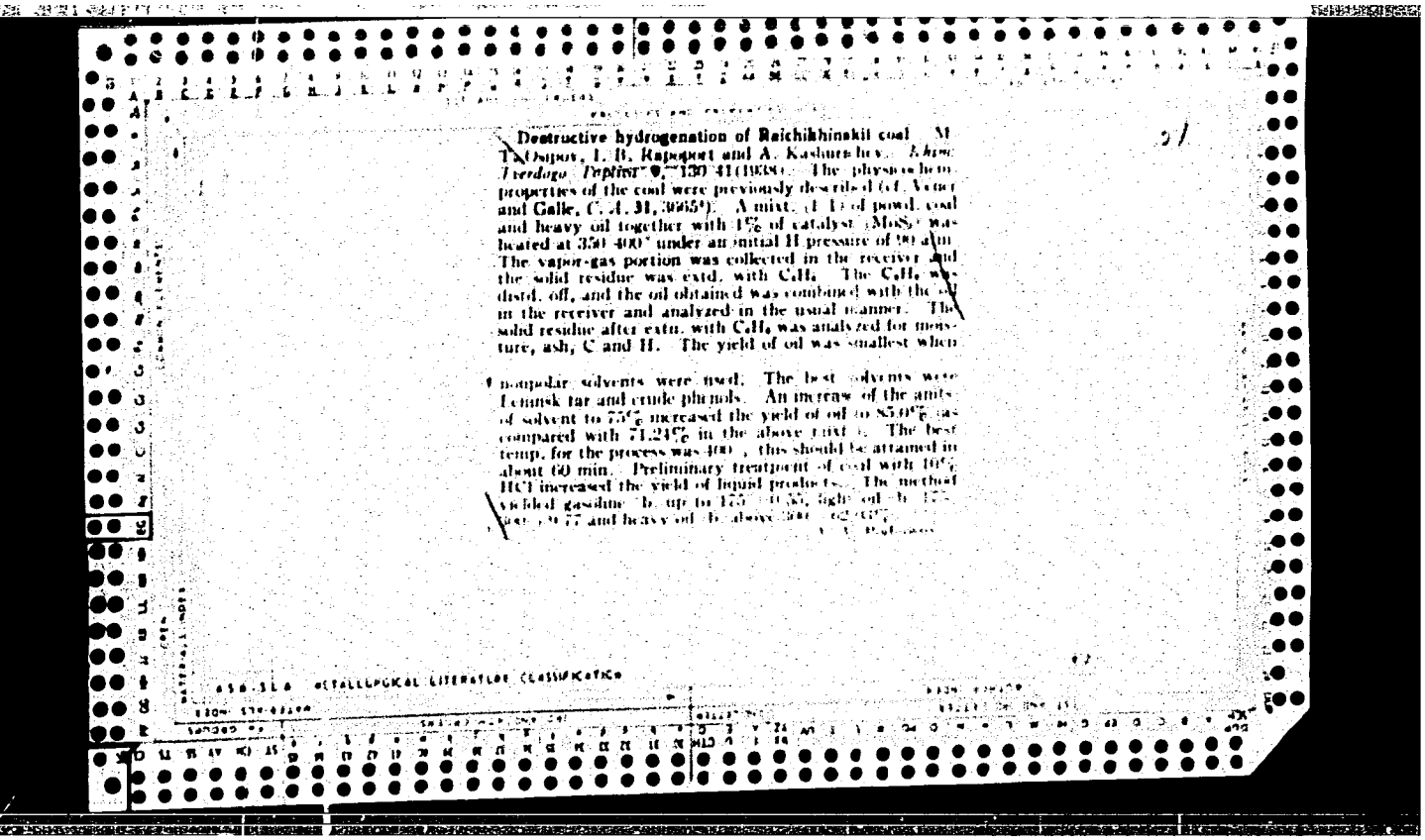


Hydrogenation over alloy catalysts at elevated hydrogen pressure. I. Hydrogenation of some aromatic compounds. I. B. Rapoport and E. I. Sil'chenko. *J Applied Chem.* (U. S. S. R.) **10**, 1427–34 (in French 1434–41(1937).—Tech. naphthalene and phenol were hydrogenated in an autoclave in the presence of various alloy catalysts at an optimal temp. of 200° and at the H_2 pressure of 100 atm. After termination of the reaction the reaction mixt. was cooled and fractionated. The yield of cyclohexane and tetrahydronaphthalene was 85-95%. An increase of the pressure increased the velocity of the hydrogenation, while the coarseness of the catalyst decreased the hydrogenation. The following catalysts were investigated: Ni-Al, Ni-Co-Al, Co-Al, Co-Si and Ni-Co-Si, with the exception of Co-Si, all gave good results. The treatment of the Ni-Co-Al catalyst with KOH yielded a more active catalyst than that treated with NaOH. Twenty-two references. A. A. Podgurny

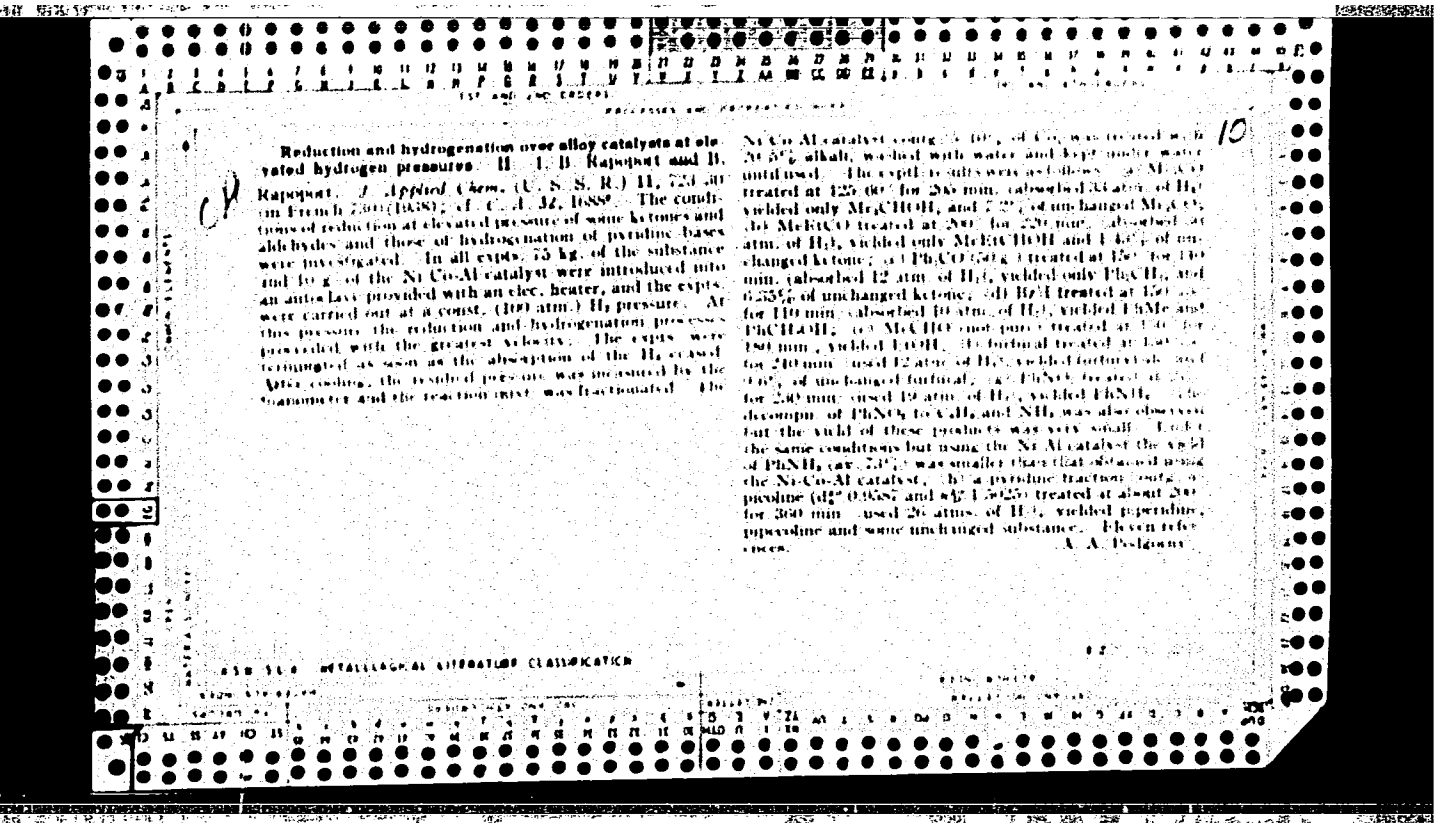


Synthesis of hydrocarbons from carbon oxide and hydrogen over alloyed catalysts II. I. B. Rapoport and I. Pokrzhitskaya. *Khim. Tverdogo Topliva*, 9 [6] 25 (1938). Pressed catalysts, 1 g. 20, 30/1, 32, loose and *Fuel Science Tech.* 8, 215–16, 216 (1938). The gas mixt. in an apparatus (CO 67, H 33%, CO₂ 0.6, O 0.2 and N 4.9%) after purification and drying was passed into a quartz tube filled with a catalyst, from where it passed into a graduated receiver, cooled with ice, into a charcoal scrubber and then into a gasometer. A portion of the gas was withdrawn for the analysis. The space rate was kept at 100–120. Ni-Al, Co-Al and Co-Si catalysts were used in the powd. and granular forms. The Ni-Al catalyst reduced in the H atm. at 350° yielded 160 cc. per cu. m. of gas of hydrocarbon under the optimal conditions and velocity 100 cc./hr. The Co-Al catalyst reduced with H in the NH₃ atm. at 220° yielded 150 cc. per cu. m. of gas hydrocarbon at 200° and vol. velocity of 50. The Co-Si catalyst prepared as the Co-Al catalyst yielded 60 cc. per cu. m. of gas hydrocarbon at 220° and vol. velocity of 50. All the above data are given for the catalysts in the granular forms; the yield of hydrocarbon using powd. catalysts was considerably lower.

V. V. Podgorny

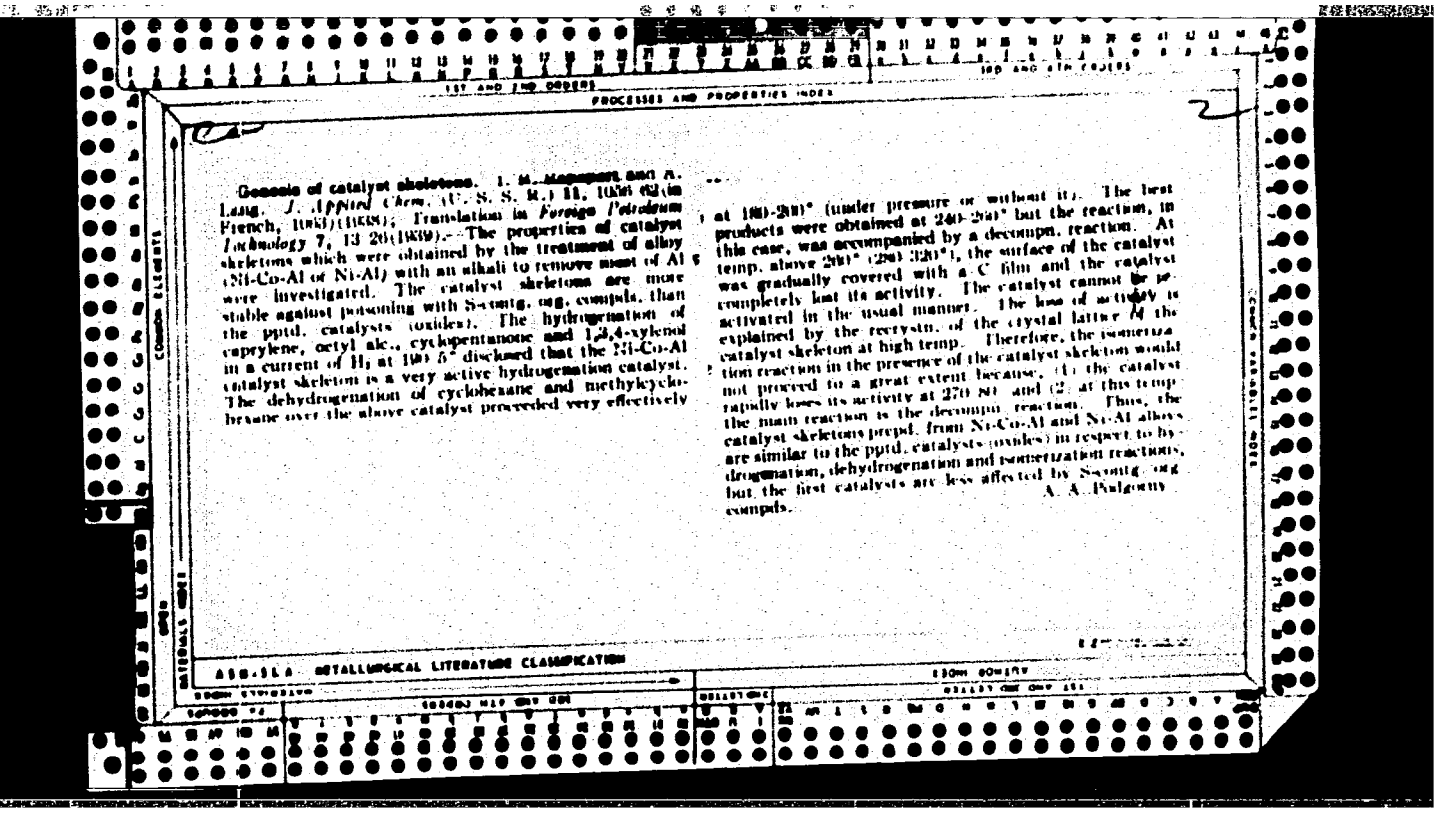


Destructive hydrogenation of Raichikhinskii coal. M. T. Osipov, L. B. Rapoport and A. Kashurnikov. *Khim. Tverdago Topliva* 9, 120-41(1938). The physicochem. properties of the coal were previously described (cf. Vener and Galle, C. A. 31, 9665[9]). A mixt. (1:1) of pond. coal and heavy oil together with 1% of catalyst (MoS_2) was heated at 350-400° under an initial H pressure of 90 atm. The vapor-gas portion was collected in the receiver and the solid residue was extd. with C_6H_6. The C_6H_6 was distd. off, and the oil obtained was combined with the oil in the receiver and analyzed in the usual manner. The solid residue after extn. with C_6H_6 was analyzed for moisture, ash, C and H. The yield of oil was smallest when nonpolar solvents were used. The best solvents were Lenansk tar and crude phenols. An increase of the amt. of solvent to 75% increased the yield of oil to 85.0% as compared with 71.24% in the above mixt. The best temp. for the process was 400°, this should be attained in about 60 min. Preliminary treatment of coal with 10% HCl increased the yield of liquid products. The method yielded gasoline (b. up to 175°) 6.5%, light oil (b. 175-[illegible]°) 9.77 and heavy oil (b. above [illegible]°) [illegible]%.



Reduction and hydrogenation over alloy catalysts at elevated hydrogen pressures II. I. B. Rapoport and B. Rapoport. *J. Applied Chem.* (U. S. S. R.) II, 723-30 (in French 730) (1938); cf. *C. A.* 32, 1088⁹. The conditions of reduction at elevated pressure of some ketones and aldehydes and those of hydrogenation of pyridine bases were investigated. In all expts. 75 kg. of the substance and 10 g. of the Ni-Co-Al catalyst were introduced into an autoclave provided with an elec. heater, and the expts. were carried out at a const. (100 atm.) H_2 pressure. At this pressure the reduction and hydrogenation processes proceeded with the greatest velocity. The expts. were terminated as soon as the absorption of the H_2 ceased. After cooling, the residual pressure was measured by the manometer and the reaction mixt. was fractionated. The Ni-Co-Al catalyst contg. 50% of Co was treated with 20-5% alkali, washed with water and kept under water until used. The expil. results were as follows: a) MeAcO treated at 125-60° for 5½ min. absorbed 34 atm. of H_2, yielded only Me_2CHOH, and 7.2% of unchanged $MeAcO$; b) MeBzCO treated at 80° for 220 min. absorbed 20 atm. of H_2, yielded only $MeBzCHOH$ and 14.6% of unchanged ketone; c) Ph_2CO (50 g.) treated at 85° for 100 min. absorbed 12 atm. of H_2, yielded only Ph_2CH_2 and 6.25% of unchanged ketone; d) BzH treated at 150° for 110 min. absorbed 10 atm. of H_2, yielded $PhMe$ and $PhCH_2OH$; e) MeCHO not pure, treated at 140° for 180 min., yielded EtOH. f) furfural treated at 140° for 240 min. used 12 atm. of H_2, yielded furfuryl alc. and 6% of unchanged furfural. g) $PhNO_2$ treated at 85° for 240 min. used 19 atm. of H_2, yielded $PhNH_2$. The decomp. of $PhNO_2$ to CaH_6 and NH_3 was also observed but the yield of these products was very small. Under the same conditions but using the Ni-Al catalyst the yield of $PhNH_2$ (at. 50%) was smaller than that obtained using the Ni-Co-Al catalyst. h) a pyridine fraction (mostly α-picoline (d_4^{20} 0.945 and n_D^{20} 1.5025) treated at about 200° for 300 min. used 26 atms. of H_2, yielded piperidine, piperidine and some unchanged substance. Eleven references.

A. A. Boldyrev

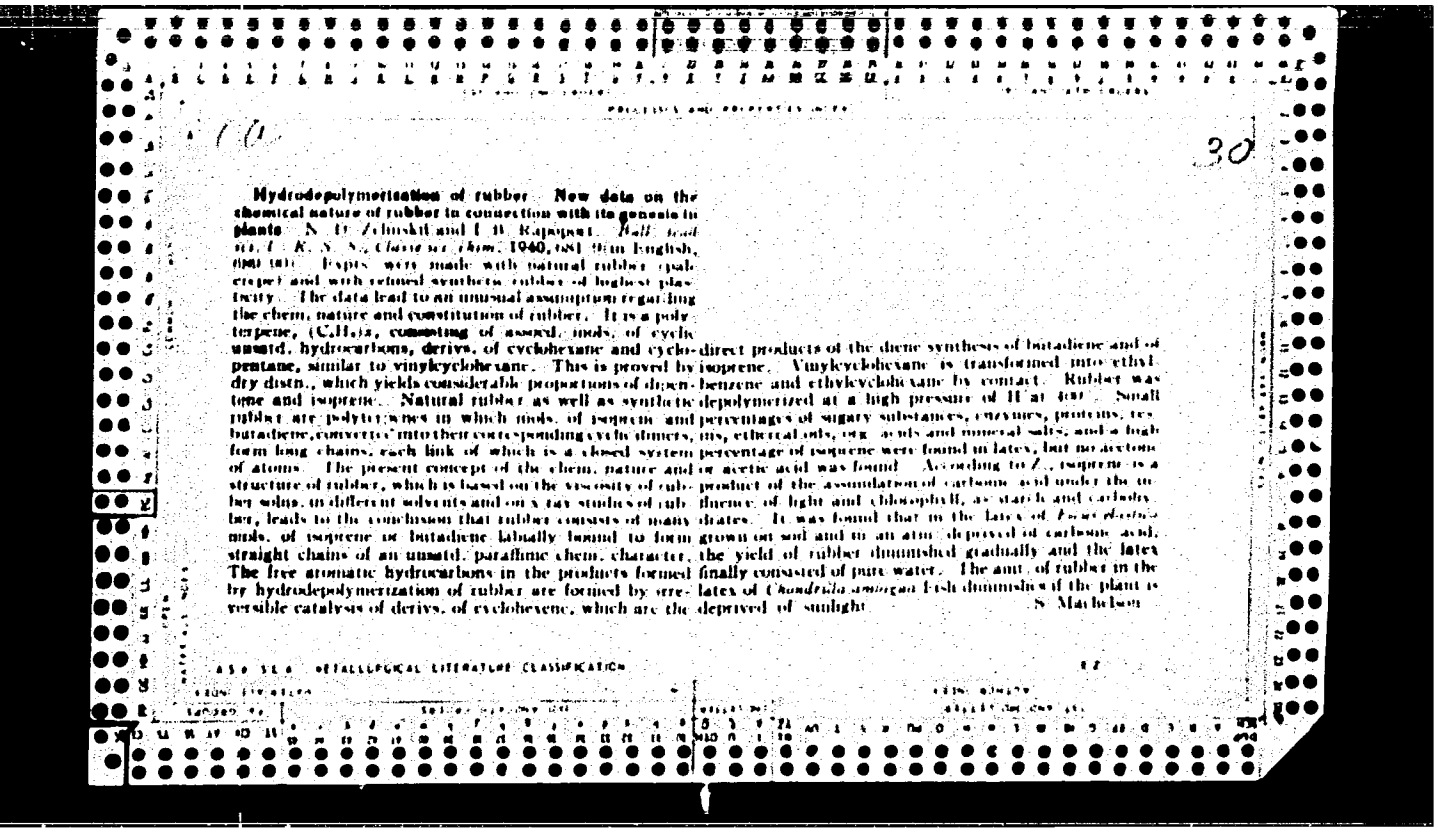


PROCESSES AND PROPERTIES INDEX

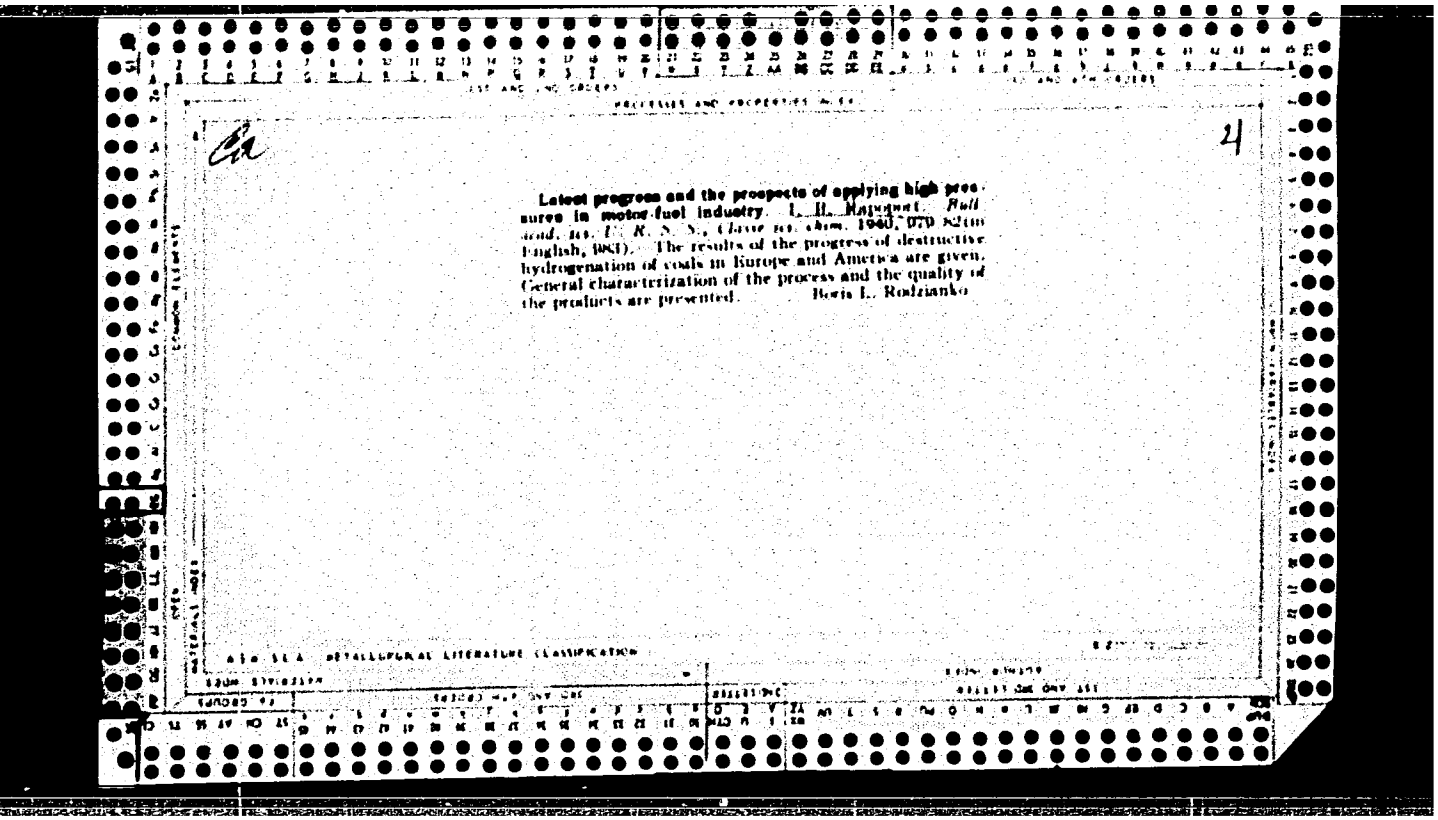
Genesis of catalyst skeletons. I. M. Magat and A. Lang. *J. Applied Chem.* (U. S. S. R.) 11, 1261-62 (in French, 1938)(1163-5); Translation in *Foreign Petroleum Technology* 7, 13-20(1939).—The properties of catalyst skeletons which were obtained by the treatment of alloy (Ni-Co-Al or Ni-Al) with an alkali to remove most of Al were investigated. The catalyst skeletons are more stable against poisoning with S-containg. org. compds. than the pptd. catalysts (oxides). The hydrogenation of propylene, octyl alc., cyclopentanone and 1,3,4-xylenol in a current of H_2 at 100-5° disclosed that the Ni-Co-Al catalyst skeleton is a very active hydrogenation catalyst. The dehydrogenation of cyclohexane and methylcyclohexane over the above catalyst proceeded very effectively at 180-200° (under pressure or without it). The best products were obtained at 260-280° but the reaction, in this case, was accompanied by a decompn. reaction. At temp. above 280° (290-320°), the surface of the catalyst was gradually covered with a C film and the catalyst completely lost its activity. The catalyst cannot be reactivated in the usual manner. The loss of activity is explained by the recrystn. of the crystal lattice of the catalyst skeleton at high temp. Therefore, the isomerization reaction in the presence of the catalyst skeleton would not proceed to a great extent because, (1) the catalyst rapidly loses its activity at 270-80° and (2) at this temp. the main reaction is the decompn. reaction. Thus, the catalyst skeletons prepd. from Ni-Co-Al and Ni-Al alloys are similar to the pptd. catalysts (oxides) in respect to hydrogenation, dehydrogenation and isomerization reactions, but the first catalysts are less affected by S-containg. org. compds.

A. A. Balandin

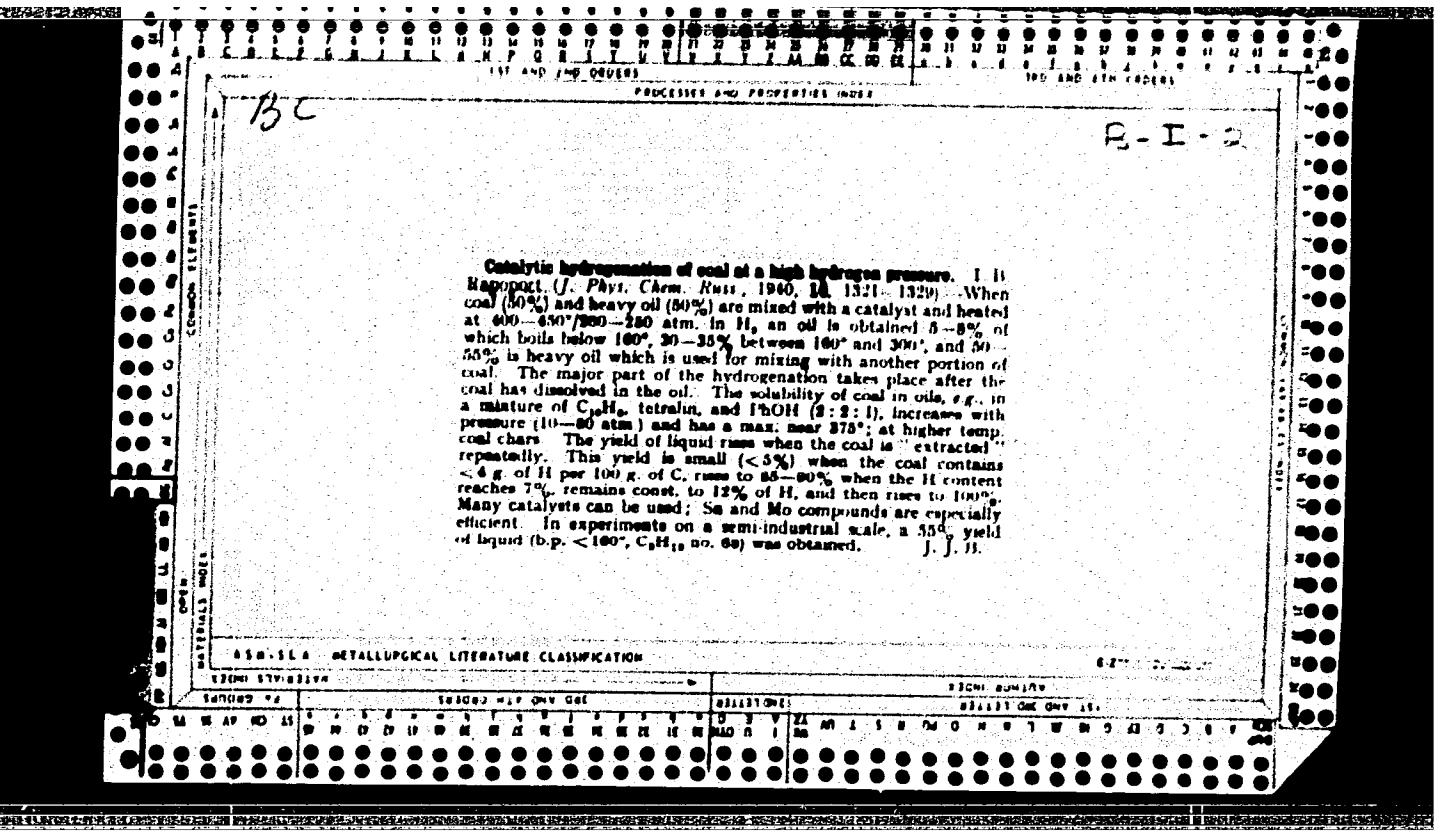
A50-81A METALLURGICAL LITERATURE CLASSIFICATION



Hydrodepolymerization of rubber. New data on the chemical nature of rubber in connection with its genesis in plants. N. D. Zelinskiĭ and I. D. Rapoport. *Bull. Acad. sci. U. R. S. S., Classe sci. chim.* 1940, 681–91 (in English, 691–701). Expts. were made with natural rubber (pale crepe) and with refined synthetic rubber of highest plasticity. The data lead to an unusual assumption regarding the chem. nature and constitution of rubber. It is a polyterpene, $(C_5H_8)_x$, consisting of assocd. mols. of cyclo-unsatd. hydrocarbons, derivs. of cyclohexane and cyclo-pentane, similar to vinylcyclohexane. This is proved by isoprene. Vinylcyclohexane is transformed into ethyl-dry distn., which yields considerable proportions of diphen-benzene and ethylcyclohexane by contact. Rubber was tene and isoprene. Natural rubber as well as synthetic depolymerized at a high pressure of H at 400°. Small rubber are polyterpenes in which mols. of isoprene and percentages of sugary substances, enzymes, proteins, res- butadiene, converts into their corresponding cyclic dimers, ins, ethereal oils, org. acids and mineral salts; and is built form long chains, each link of which is a closed system percentage of isoprene were found in latex, but no acetone of atoms. The present concept of the chem. nature and of acetic acid was found. According to Z., isoprene is a structure of rubber, which is based on the viscosity of rub- product of the assimilation of carbonic acid under the in- luence of light and chlorophyll, as starch and carbohy- ber solns. in different solvents and on x-ray studies of rub- drates, leads to the conclusion that rubber consists of many mols. of isoprene or butadiene usually found to form grown on soil and in an atm. deprived of carbonic acid, straight chains of an unsatd. paraffinic chem. character, the yield of rubber diminished gradually and the latex The free aromatic hydrocarbons in the products formed finally consisted of pure water. The amt. of rubber in the by hydrodepolymerization of rubber are formed by irre- latex of *Chondrilla aurigeus* fell diminished if the plant is versible catalysis of derivs. of cyclohexene, which are the deprived of sunlight. S. Matchett



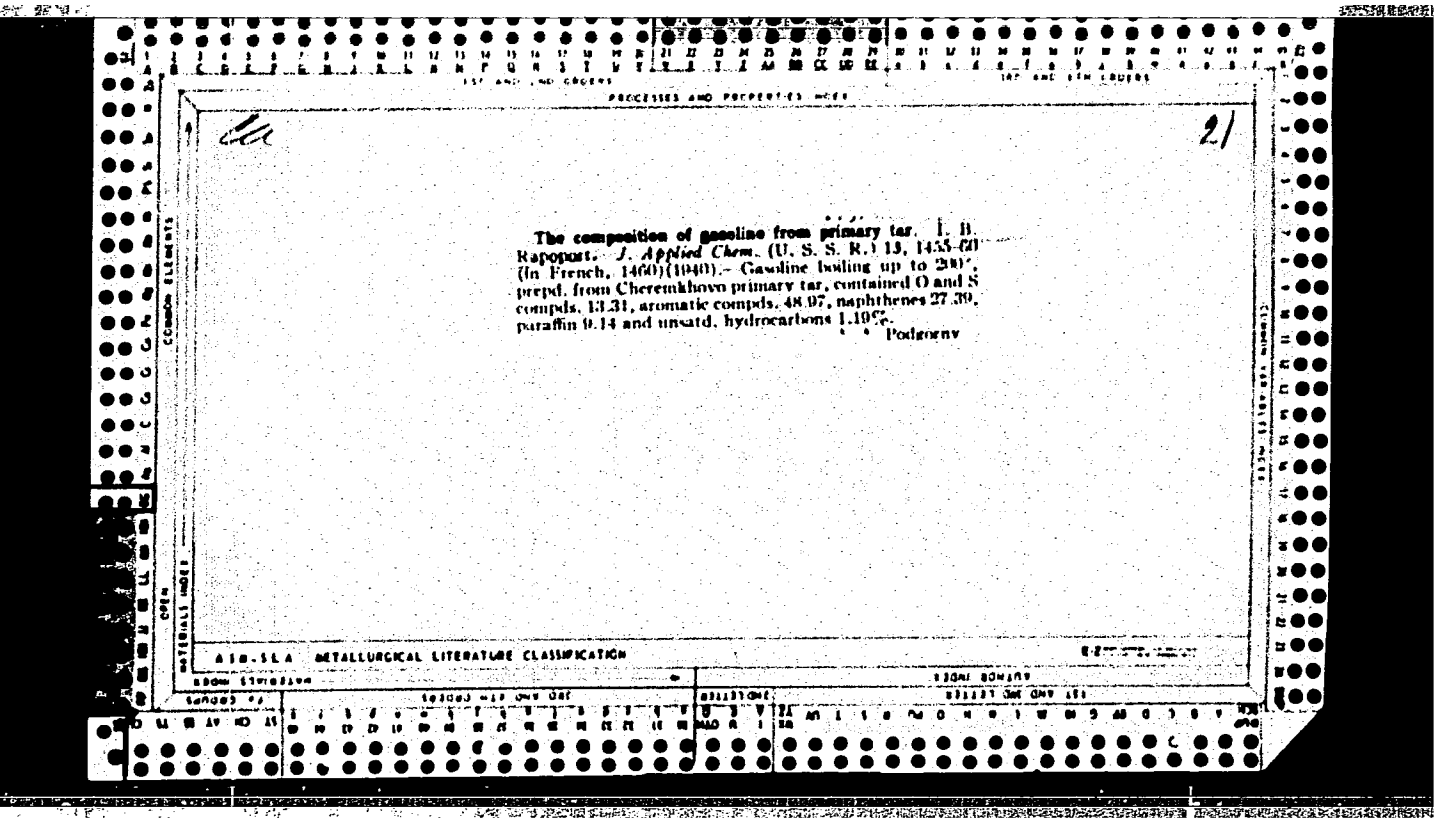
Latest progress and the prospects of applying high pressures in motor-fuel industry. L. H. Karpenet. *Bull. acad. sci. U. R. S. S., Classe sci. chim.* 1940, 079-82(in English, 083). The results of the progress of destructive hydrogenation of coals in Europe and America are given. General characterization of the process and the quality of the products are presented. Boris L. Rodzianko



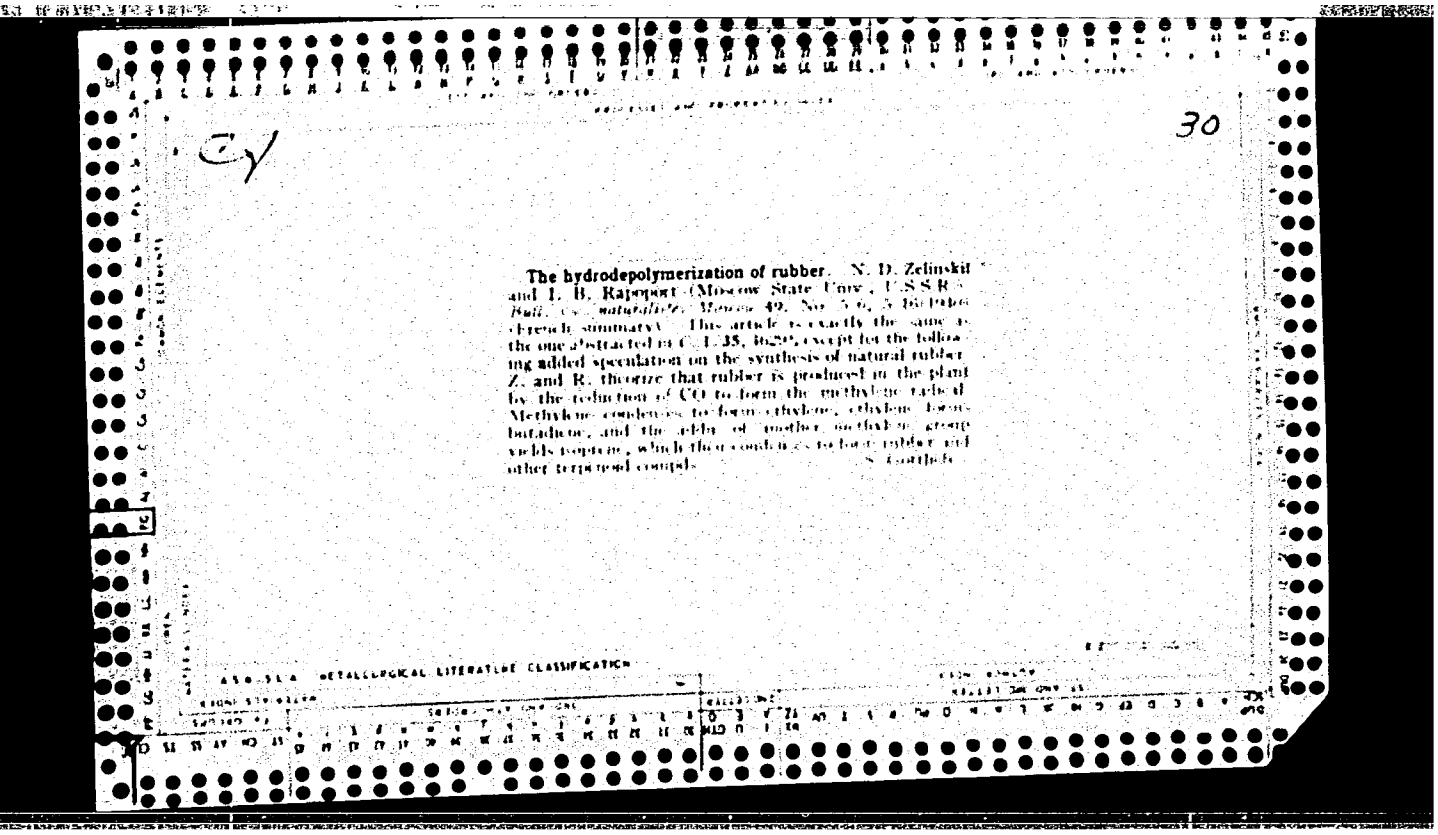
BC

B-I-2

Catalytic hydrogenation of coal at a high hydrogen pressure. I. I. Rapoport. (*J. Phys. Chem. Russ.*, 1960, **34**, 1321–1329).—When coal (50%) and heavy oil (50%) are mixed with a catalyst and heated at 400—450°/350—250 atm. in H_2, an oil is obtained 5—8% of which boils below 160°, 30—35% between 160° and 300°, and 50—55% is heavy oil which is used for mixing with another portion of coal. The major part of the hydrogenation takes place after the coal has dissolved in the oil. The solubility of coal in oils, *e.g.*, in a mixture of C_6H_6, tetralin, and PhOH (2 : 2 : 1), increases with pressure (10—60 atm.) and has a max. near 375°; at higher temp. coal chars. The yield of liquid rises when the coal is "extracted" repeatedly. This yield is small (< 5%) when the coal contains < 4 g. of H per 100 g. of C, rises to 85—90% when the H content reaches 7%, remains const. to 12% of H, and then rises to 100%. Many catalysts can be used; Se and Mo compounds are especially efficient. In experiments on a semi-industrial scale, a 55% yield of liquid (b.p. < 180°, C_nH_{2n} no. 60) was obtained. J. J. H.



The composition of gasoline from primary tar. I. B. Rapoport. *J. Applied Chem.* (U. S. S. R.) 13, 1455-60 (In French, 1460)(1940).- Gasoline boiling up to 200°, prepd. from Cheremkhovo primary tar, contained O and S compds. 13.31, aromatic compds. 48.97, naphthenes 27.39, paraffin 9.14 and unsatd. hydrocarbons 1.19%.
Podgorny



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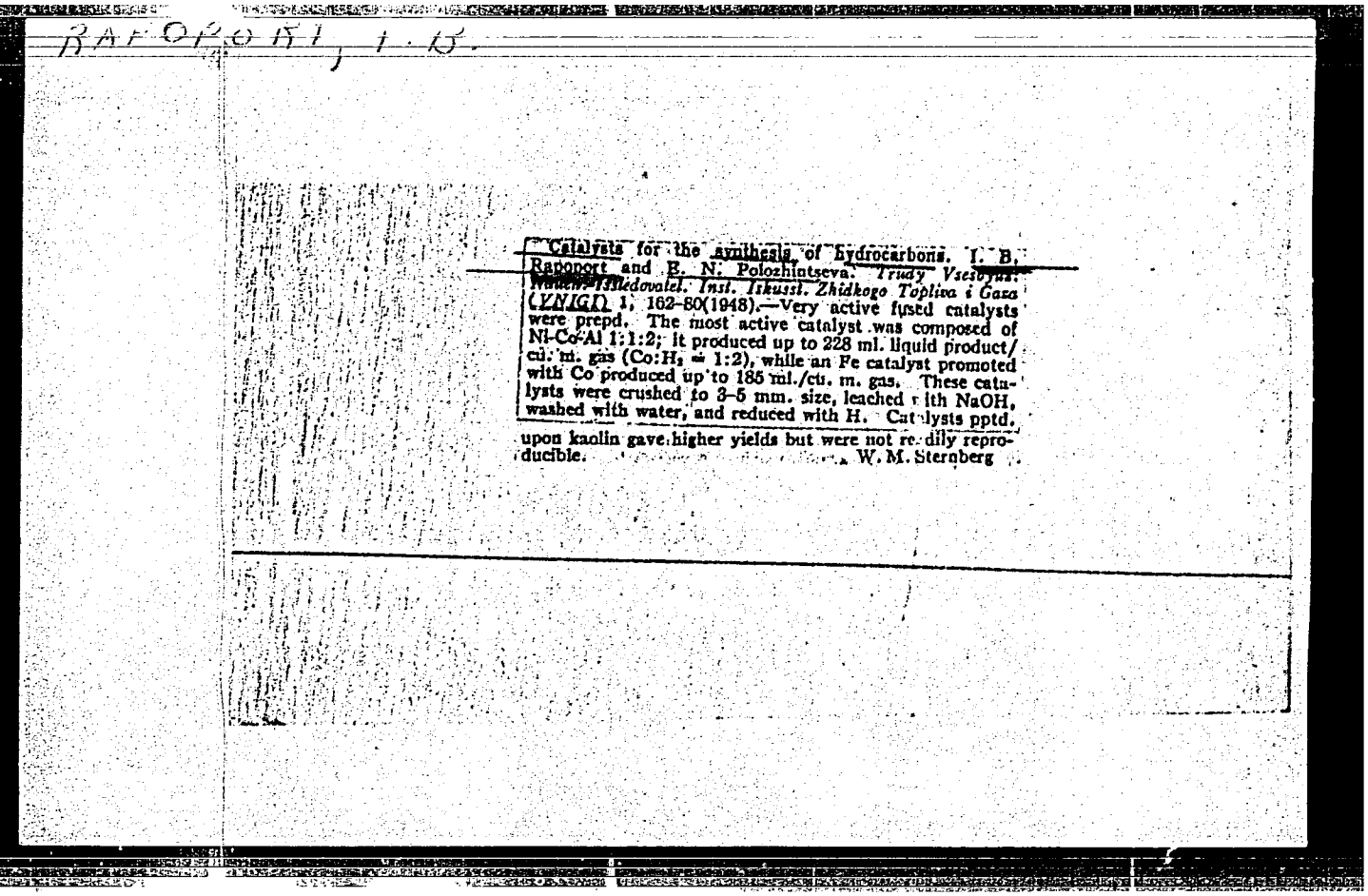
The hydrodepolymerization of rubber. N. D. Zelinskiĭ and I. B. Rapoport (Moscow State Univ., U.S.S.R.). *Bull. Soc. naturalistes Moscou* 49, No. 5-6, 3 (1940) (French summary). This article is exactly the same as the one abstracted in C. A. 35, 8028, except for the following added speculation on the synthesis of natural rubber. Z. and R. theorize that rubber is produced in the plant by the reduction of CO to form the methylene radical. Methylene condenses to form ethylene, ethylene forms butadiene, and the addn. of another methylene group yields isoprene, which then condenses to form rubber and other terpenoid compds. S. Gottlieb

A.S.T.M. METALLURGICAL LITERATURE CLASSIFICATION



BAROKH, I. B.

Catalysts for the synthesis of hydrocarbons. I. B. Rapoport and E. N. Polozhintseva. *Trudy Vsesoyuz. Nauch.-Issledovatel. Inst. Iskusst. Zhidkogo Topliva i Gaza* (*VNIGI*) 1, 162–80(1948).—Very active fused catalysts were prepd. The most active catalyst was composed of Ni-Co-Al 1:1:2; it produced up to 228 ml. liquid product/cu. m. gas (Co:H_2 = 1:2), while an Fe catalyst promoted with Co produced up to 185 ml./cu. m. gas. These catalysts were crushed to 3–5 mm. size, leached with NaOH, washed with water, and reduced with H_2. Catalysts pptd. upon kaolin gave higher yields but were not readily reproducible. W. M. Sternberg

Rapoport, L. and Kozina, M. - "Polymerization of [illegible] boron fluoride", (In Union: 1 August: A. Rapoport, Irudy Mask. In-ta [illegible] khim. tekhnologii im. Lomonosova, Issue 2, 1949, p. 69-89, - Bibliog: 15 items.

SO: U-3042, 11 March 53, (Letopis 'Zhurnal 'nykh Statey, No. 8, 1949)

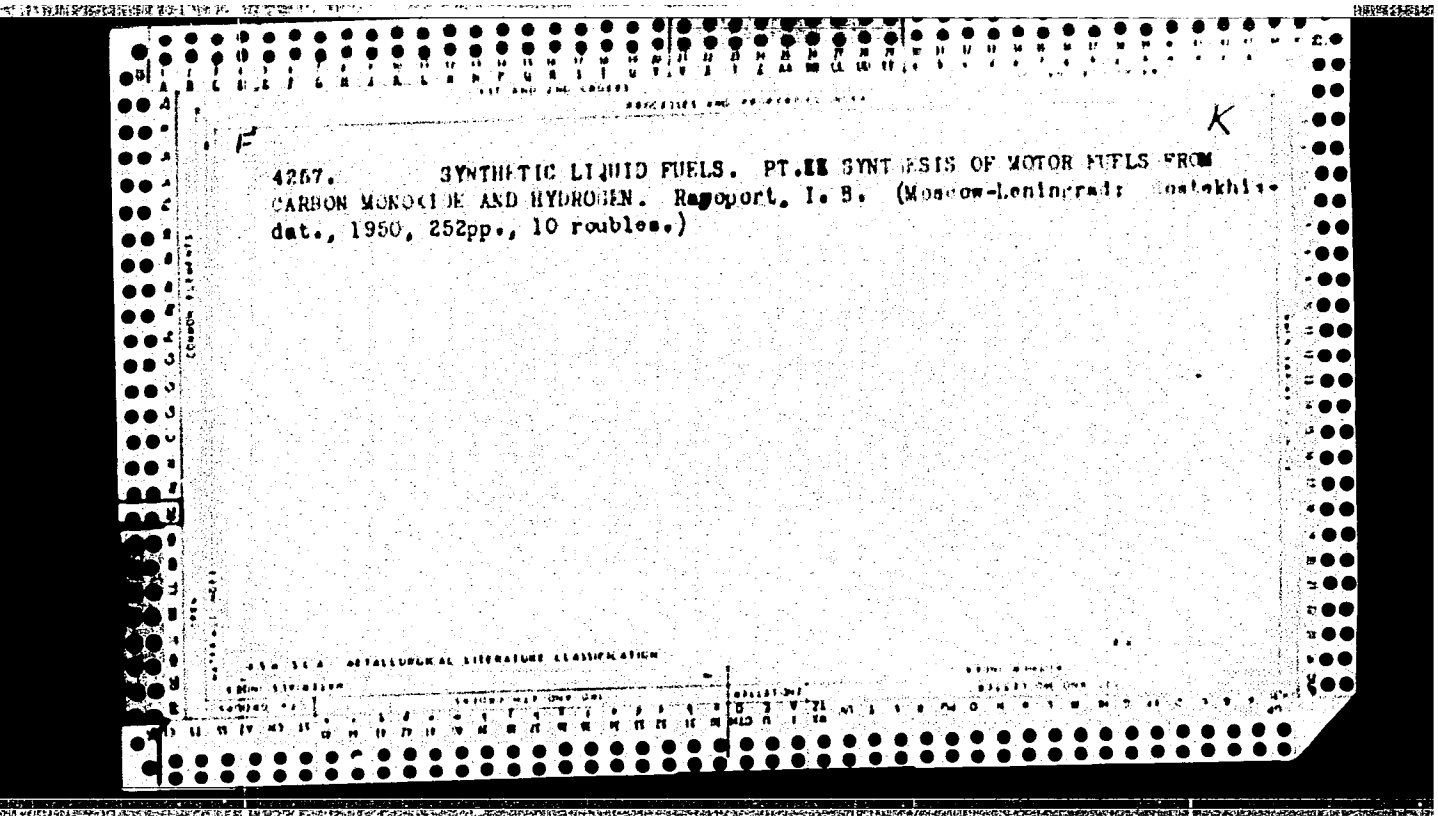
F.A

X

1489. SYNTHETIC LIQUID FUELS. PT I. HYDROGENATION OF FUELS. (ISKUSSTVENNOE ZHIDKOE TOPLIVO CHAST I. GIDROGENIZATSIIA TOPLIV). Report, I.B. (Moscow and Leningrad: Govt Sci. Tech. Publ. Ho. for Petrol. Mineral-Fuel Lit., 1949, 332pp.).

Presents cases of chemistry and technology of destructive hydrogenation of liquid and solid fuels. Influences of temperature, pressure, catalysts, and solvent on the process of destructive hydrogenation are thoroughly reviewed on the basis fo the literature and the author's experiences. Treatment o f the products of hydrogenation for production of gasoline is comprehensively described. Recommended for use of advanced students in the fields of petroleum and chemical engineering, or as a handbook. References follow each chapter.

B.L.R.



4267. SYNTHETIC LIQUID FUELS. PT.II SYNTHESIS OF MOTOR FUELS FROM CARBON MONOXIDE AND HYDROGEN. Rapoport, I. B. (Moscow-Leningrad: Gostekhiz-dat., 1950, 252pp., 10 roubles.)

RAPOPORT, I. B. - LEVKOVICH, M. M.

Hydrocarbons

Mechanism of synthesis of hydrocarbons from carbon monoxide and hydrogen over iron catalysts. Dokl. AN SSSR 84, no. 4, 1952.

Monthly List of Russian Accessions, Library of Congress, October, 1952. Unclassified.

RAPOPORT, I. B.

FAL'KOVSKAYA, A.A.; RAPOPORT, I.B.

Synthesis of hydrocarbons with iron catalysts; survey of literature.
Trudy VNIGI no.6:60-84 '54. (MLRA 7:11)
(Hydrocarbons) (Catalysts)

USSR/Chemical Technology. Chemical Products and Their Application -- Treatment of natural gases and petroleum. Motor fuels. Lubricants, I-13

Abst Journal: Referat Zhur - Khimiya, No 2, 1957, 5554

Author: Kheyfets, Ye. M., Milovidova, N. V., Borukhova, M. S., Rapoport, I. B.

Institution: None

Title: Investigation of the Products of Synthesis from CO and H_2 Over Iron Catalysts

Original Publication: Khimiya i tekhnologiya topliva, 1956, No 5, 8-17

Abstract: Description o fthe results of an investigation of the products of synthesis from CO and H_2 over a Fe-Cu catalyst activated with borax and potash (K-1) and over a Fe catalyst activated with potash (K-2). Syntheses over K-1 and K-2 were conducted, respectively, at a pressure of 10 and 30 atmospheres, at temperatures of 200-250° and ~300°, space velocity of ~80 $hour^{-1}$ and ~1,000 $hour^{-1}$, in a pilot-plant and in a semi-production scale unit, with reactors of different holding

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RAPPOPORT, I. B.

602. COMPOSITION AND PROPERTIES OF SYNTHETIC PRODUCTS FROM CARBON MONOXIDE AND HYDROGEN WITH AN IRON CATALYST. I. Kheifets, B.H., Milovidova, N.V. and Rapoport, I.B. (Khim. Tekhnol. Topliva (Chem. Technol. Fuel, Moscow), 1956, (2), 35-45; abstr. in Chem. Abstr., 1956, vol. 50, 11643).

Fuel 3

The pilot plant synthesis with iron-copper catalyst promoted with potassium silicate was effected under a pressure at 10 atm, at 210-40°, and a space velocity of 80-100/hour. Fractional distillation, chromatography, formation of complexes with urea, fractional crystallation from ethylmethylketone, reaction with antimony chloride and spectroscopic examination showed that the products consist of saturated and unsaturated hydrocarbons mostly of normal structure with a small amount of oxygen-containing compounds (3.5% acids, ethers, alcohols, aldehydes, ketones). The amount of unsaturated hydrocarbons in separate fractions decreases with increase in molecular weight; they consist of trans isomers (50-70%) and α -olefins. No other isomers, cyclic, or resin compounds are present. Significant amounts of unsaturated hydrocarbons in the product permits its use for manufacture of alcohols, acids, detergents, and solvents. Fractions to 180° can be used as motor fuel; fractions 180-320° as high cetane addition to diesel fuel. C.A.

USSR/Kinetics - Combustion. Explosions. Topochemistry. Catalysis. B-9

Abs Jour : Referat Zhur - Khimiya, No 6, 1957, 18632

Author : B.P. Vaynshteyn, Ye.A. Plokhinskaya, I.B. Rapoport.
Title : Influence of Alkaline Additions on Activity and Selectivity of Iron-Copper Catalysts. Report I.

Orig Pub : Khimiya i tekhnol topliva, 1956, No 8, 31-35

Abstract : The activity of precipitated iron-copper catalysts (2 to 10% of Cu) in the synthesis reaction of hydrocarbons of CO and H_2 depending on the content of added silicate compounds (I) or MgO in the catalysts was investigated at 212 to 218° and the gauge pressure of 10 atm. The yield of oil and paraffins contained in the gases, if the addition of I was increased by 10 to 20%. Catalysts containing 2 to 5% of Cu and 15 to 20% of I showed a high activity and stability (2800 hours) and yielded a higher amount of liquid and solid hydrocarbons than catalysts with a greater Cu content. Introduction of MgO instead

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Rapoport I.B.

2573. PRODUCTS OF REACTION OF CARBON MONOXIDE AND HYDROGEN WITH IRON CATALYSTS. Khalfoin, E.M., Ilovaiskova, A.V., Borukhova, N.S. and Rapoport, I.B. (Khim. Tekhnol. Topliva (Chem. Technol. Fuel, Moscow), 1956, (5), 8-17; abstr. in Chem. Abstr., 1956, vol. 50, 15048). Reaction on a semi-industrial scale of carbon monoxide and hydrogen over an iron-copper catalyst activated by borax and potassium carbonate at 200-260° under 10 atm pressure, and 80 h^{-1} volume rate gave a fraction boiling up to 320° in which alcohols constituted 16-30% of the oxygen-containing compounds, 30-40% had branched structure, and the rest of the oxygen-containing fraction consisted of aldehydes, ketones, and acids. Hydrocarbons, mostly α-olefins, comprised 55-8[illegible] of the yield. With potassium carbonate-activated iron catalyst at 3000 under 30 atm pressure and 1000 h^{-1} volume rate, the yield of α-olefins increased to 80-87% and the oxygen-containing fraction contained 4.8% alcohols, 10.4% ethers, 0.2% acids, and some aldehydes and ketones. The industrial aspects of this synthesis are briefly discussed. C.A.

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541

The influence of organic compounds of sulphur on the synthesis process over iron catalysts. (Cont.)

15-20 g/m^3). The loss of activity was observed for both purified and unpurified gas and was due to the precipitation of paraffin. On extraction of paraffin the activity of the catalyst was regenerated. Similar experiments at a pressure of 10 atm indicated that the activity of the catalyst was not affected after 60 days of continuous operation on both kinds of synthesis gas. Some of the experimental results and material balances of the process are given in tables. It was shown that the catalyst reacts with organic sulphur compounds which decompose with the formation of metallic sulphides. The products of the synthesis did not contain sulphur compounds. 3 figures, 5 tables. There are six references, all non-Russian.

Card 2/2

RAPOPORT, I.B.; MUZYKANTAYA, O.A.

Effect of organic sulfur compounds on the process of synthesis over iron catalysts. Khim.i tekh.topl.i masel no.5:19-27 My '57. (MIRA 10:7)

1. Vsesoyuznyy nauchno-issledovatel'skiy institut neftyanoy promyshlennosti.
(Catalysts) (Sulfur compounds)

Paraffins from sulphurous crude oils as a raw material for the production of synthetic fatty acids. (Cont.) 65-6-7/13

permanganate as a catalyst (0.2-0.3%) by air (120 l/kg/hr); washing of the oxidation products with water, saponification with NaOH; separation of unsaponified product I (unsaponified in an autoclave at 180-185 C and 9 atm), separation of unsaponified product II (thermal treatment at a high or low pressure: t = 320-350 C, p = 120-130 atm, or t = 360-375 C; p = 3-5 atm) the decomposition of soaps with sulphuric acid, washing with water and distillation. Results of oxidation of paraffin from a distillate (370-500 C) from a mixture of sulphurous crudes are given in table 2, characteristics of fatty acids produced - table 3; yield of oxidation products - table 4, results of oxidation of paraffin at a higher temperature (125-107 C) - table 5. It was established that purified paraffin (containing up to 2% of oil and up to 0.1% of sulphur) produced from a distillate boiling at 370-500 C from a mixture of sulphurous crude oils is suitable for oxidation into synthetic fatty acids which can be used in soap making. Technical fatty acids produced leave up to 43-45% of residue on distillation which is about 24% of the starting material as against 15.5% for corresponding fatty acids from the Drogobych paraffin. The yield of the

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RAPOPORT, I. B.

398. INFLUENCE OF ADDITION OF ALKALIS ON THE ACTIVITY AND SELECTIVITY OF IRON-COPPER CATALYSTS. Vainshtein, B.P., Ploldinskaya, E.A. and Rapoport, I.B. (Khim. Tekhnol. Topliva (Chem. Technol. Fuel, Moscow), 1956, (8), 31-35; abstr. in Chem. Abstr., 1957, vol. 51, 2250). Potassium silicates were added to Fischer-Tropsch synthesis catalysts at 10 atm and temperatures of 212-218°, with carbon monoxide:hydrogen ratios of 1:0.70. With increase of silicate addition to catalysts containing 2-10% copper, yields of oils and heavy paraffin hydrocarbons are raised. An activated iron-copper catalyst reduced under hydrogen containing 5% copper and 17.5% silica gives a 50% yield of heavy paraffin hydrocarbons boiling above 300°, containing 3 or more carbon atoms. After long use the catalyst loses activity and selectivity, and the yield decreases about 20% in 3000 hours. Addition of magnesia raises the yield of the fractions boiling at 200-300° and 300-450° at the expense of the yields of the fractions boiling above 450°. C.A.

RAPOPORT, I.B.; FLID, R.M.; LIS, K.

Polymerization and cyclization of isobutylene. Dokl. AN SSSR 116 no.2: 244-247 S '57. (MIRA 11:2)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V. Lomonosova. Predstavleno akademikom B.A. Kazanskim.
(Polymerization) (Propene)

KAPOYCKI, I. B.

AUTHORS: Rapoport, I. B., Flid, R. M., and Lis, K. 20-2-23/50

TITLE: On the Polymerization and Cyclization Reactions of Isobutylene (O reaktsii polimerizatsii i tsiklizatsii izobutilena).

PERIODICAL: Doklady AN SSSR, 1957, Vol. 116, Nr 2, pp. 244-247 (USSR)

ABSTRACT: The polymerization of olefines with different numbers of carbon atoms takes place at not high temperatures, at elevated and non-elevated pressures and in the presence of various catalysts which possess properties of acids. Thermodynamical data on the polymerization of isobutylene show that the reaction very thoroughly takes place at 100-200°C. A rise of temperature reduces the activity of the catalyst due to its poisoning with resin products. The products of polymerization are the same on aluminum-silicate catalysts, independent of the fact whether these latter are synthetic or taken from nature. Dimers and trimers are the chief products. The speed of polymerization of isobutylene is much higher than that of butylene (according to Kazanskiy and Rozengart). The polymerization of butylene on aluminum-silicate catalysts is accompanied by an isomerization under

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formation of various isomeriv octenes. At 370°C olefinelike and at 450-500°C aromatic hydrocarbons form. Higher compounds, up to pentamers, can also form on various catalysts. According to thermodynamic calculations the aromatization reaction is also possible for isobutylene. The thermodynamic analysis made by the authors as well of the gross reaction as of its individual stages leads to the following main conclusions: 1. The gross reaction $2C_4H_8 \rightleftarrows 3H_2+C_6H_4(CH_3)_2$ (o-, m-, p-) (table 1) is possible (when P = 1 atmosphere) with a practically complete conversion of isobutylene already at lower temperatures (120-130°C) under a predominant formation of m-xylol. The equilibrium of the stage of cyclization of isooctene to isomeric (cis- and trans-, o-, m-, p-) dimethylcyclohexanes is practically independent on temperature displaced to the right. 2. The degree of dimerization of isobutylene decreases with increasing temperature, With increasing pressure the reaction is somewhat displaced to the right. 3. A equilibrium yield of a xylol-mixture in dehydrations of isomeric (cis- and trans-,

o-, m-, p-) dimethylcyclohexanes increases with increasing temperature. A full conversion can practically be attained at a temperature of 600°C (when P = 1 atmosphere). After some considerations on further possibilities and desired investigations the authors state that the present paper is dedicated to the study of the polymerization and aromatization reactions over the catalyst MoO_3-Al_2O_3. The experimental part with the usual data and conclusions follows: 1. The posssibility of a polymerization with subsequent aromatization of isobutylene over a molybdenum-aluminum-catalyst was for the first time proved. 2. At temperatures up to 200°C the polymerization reaction over the above-mentioned catalyst proceeds under formation of di-, tri- and still higher polymers. The direction of reaction changes with increasing temperature and aromatic hydrocarbons appear in the product of the catalysis. 3. In the fration boiling at 132-148°C, which had formed on the passage f isobutylene over the Mo-Al-catalyst at 400°C, up to 60% para- and meta-xylol, i.e. about 6 g per 1 m^3 gas were determined.

On the Polymerization and Cyclization Reactions of Isobutylene 69-2-23/50

There are 4 tables and 7 references, 6 of which are Slavic.

ASSOCIATION: Moscow Institute for Fine Chemical Technology imeni M. V. Lomonosov (Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova).

PRESENTED: By B. A. Kazanskiy, Academician, May 4, 1957

SUBMITTED: May 3, 1957

AVAILABLE: Library of Congress.

Card 4/4

KHEYFETS, Ye.M.; MILOVIDOVA, N.V.; ZEL'VYANSKAYA, Ye.B.; LL'IN, B.I.; YUDAKOVA, R.N.; RAPOPORT, I.B.

Production of detergents from olefins. Khim. i tekhn.topl. i masel 3 no.48-54 S '58. (MIRA 11:10)

1. Vsesoyuznyy nauchno-issledovatel'skiy institut po pererabotke nefti i gaza i polucheniyu iskusstvennogo zhidkogo topliva.
(Cleaning compounds) (Olefins)

RAPOPORT, I.B.; KRUGLIKOV, V.Ya.; BOL'SHOV, I.I.

Developing a highly productive process of synthesizing compounds from CO and H_2. Khim. i tekh. topl. i masel 3 no.12:36-41 D '58. (MIRA 11:12)

1. Vsesoyuznyy nauchno-issledovatel'skiy institut neftyanoy promyshlennosti.
(Chemistry, Organic--Synthesis) (Hydrocarbons)

SOV/ 65-58-6-12/13

AUTHORS: Vaynshteyn, B. P; Rapoport, I. B. and Plokhinskaya, Ye.A.

TITLE: Investigations of Conditions for the Reduction of an Iron-Copper Catalyst. (K voprosu ob usloviyakh vosstanovleniya zhelezo-mednogo katalizatora).

PERIODICAL: Khimiya i Tekhnologiya Topliv i Masel, 1958, Nr.6. pp. 65 - 70. (USSR).

ABSTRACT: Experiments were carried out and results are given for the reduction of precipitated iron-copper catalysts which are used for the synthesis of hydrocarbons from CO_2 and hydrogen. Reduction was carried out at volume rates of 900 - 6,000 hours^{-1} when the reduction was carried out with synthesis gas from 0.5 - 36 hours, and with hydrogen at volume rate of 3,000 hours^{-1} for varying lengths of time. Investigations were also carried out on the dynamics of decomposition of hydrocarbonates, carbonates and hydrates of metals of the catalyst when heating up to a temperature of reduction. The latter experiments were carried out in conjunction with I. V. Malyshevaya. The catalysts were heated up to reduction temperatures in a current of synthesis gas, hydrogen or nitrogen. It was found that the maximum quantity of CO_2 was separated at 170° - 180°C (Fig.1). The quantity of water

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Investigations of Conditions for the Reduction of an Iron-Copper Catalyst.

separated during the heating of the catalyst to 230°C in a current of hydrogen or nitrogen was shown to be practically equal (Fig.2). It can be observed that the activity of samples of the catalyst heated up to the temperature of reduction in a current of a gas mixture and nitrogen decreases (Fig.3). The effect of the time of reduction of the catalyst in a current of synthesis gas on its activity was investigated. When the time of reduction was increased from 0.5 to 12 hours the yield of synthesis products increased (Fig.4). Results of the effect of various volume rates on the activity of the catalyst show that catalysts, reduced at volume rates of 1500 - 3,000 hours^{-1} were most active (Fig.5). Tables 1 and 2 give the fractional composition of the synthesis products prepared with the investigated samples of catalysts. Table 3: the dependence of the activity of the catalysts on the time and temperature of reduction at the volume rate of 3,000 hours^{-1}. It was shown that an increase in the temperature of reduction of the catalyst with hydrogen to 250°C makes it possible to reduce the length of the reduction process from 24 to 6 hours whilst maintaining the normal activity

Investigations on Conditions for the Reduction of an Iron-Copper Catalyst.

of the catalyst. Further experiments were carried out on the effect of water vapour on the activity of iron-copper catalysts. These experiments were carried out together with V. P. Khokhlov. The reduction was carried out at 250°C, at atmospheric pressure, when the volume rate of hydrogen = 3,000 hours^{-1}. It was found that the period of processing of 8 - 12 hours at 225°C gave best results. During this time the CO_2 content in the outlet gas = 28% - 34%, which corresponds to a 78% - 82% process of CO. Fig.6: the dependence of the activity of the catalyst on the moisture content in the hydrogen used during the reduction. The moisture content should not be higher than 0.2 - 0.3 g/m^3. There are 6 Figures and 3 Tables.

ASSOCIATION: VNII NP.

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SOV/65-58-9-10/18

AUTHORS: Kheyfets, Ye. E; Milovidova, N. V. Zel'yanskaya, Ye. B; Il'in, B. I; Yudakova, R. N; Rapoport, I. B.

TITLE: The Preparation of Detergents From Olefins. (Polucheniye moyushchikh veshchestv iz olefinov)

PERIODICAL: Khimiya i Tekhnologiya Topliv i Masel, 1958, Nr 9, pp 48 - 54, (USSR)

ABSTRACT: C_8 - C_{18} olefins are used as raw materials in the preparation of secondary alkyl sulphates. These compounds are marketed in the West under the trade name "Teepol". More raw materials become available when C_5 - C_8 unsaturated hydrocarbons are utilized. The latter are obtained in considerable quantities during the Fischer-Tropsch process and during the cracking of paraffin. These olefins can be polymerised to di- and trimers over Mo-and Ni-catalysts. Preliminary investigations confirmed literature data on the possibility of preparing olefins boiling between 150° - 300°C by dehydrogenation of paraffins boiling within the same limits. Thus it was possible to use paraffin obtained during the carbamide deparaffination of diesel oil for the preparation of "Teepols". Olefins obtained in this way occur in a mixture with saturated paraffins and are treated with sulphuric acid.

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The Preparation of Detergents From Olefins.

During this process dialkyl sulphates and polymerised olefins are formed (Ref.18). The yield and quality of the products is influenced by the concentration of H_2SO_4, by the molar ratio H_2SO_4-olefins, the temperature and length of the reaction, by the conditions of mixing the raw material and the reagents, and by the conditions of neutralisation and hydrolysis. This method was used for the preparation of detergents from different starting materials containing varying amounts of unsaturated hydrocarbons. Synthesis gas, cracked paraffin and dehydrogenated paraffins were used as starting materials. Their content in unsaturated hydrocarbons varied between 7 and 68% (Table 1). Process conditions were such that minimal side reactions of polymerisation and formation of dialkyl sulphates were achieved. These products were sulphonated in a glass apparatus (Fig.1), and contacted with H_2SO_4 for 20 - 70 seconds. The reaction products were neutralised with a 35% solution of NaOH and the formed dialkyl sulphates hydrolised for two hours at 70°. The unreacted hydrocarbons and formed polymers were separated from the aqueous alkyl sulphate solution by settling and extraction. They were treated with

The Preparation of Detergents From Olefins.

Na_2CO_3 and concentrated over a water bath. The final product, depending on the concentration of the active substance, appeared as a powder (containing about 20% of active substance) or as a paste (approximately 50% of active substance). Aqueous alkyl sulphate solutions of given concentration were also prepared (Ref.10) Results of tests carried out on the sulphonation of narrow fractions containing mainly C_{10}, C_{12}, C_{13} and C_{15}-C_{17} fractions are tabulated (Table 2). Table 3: data on the preparation of detergents from olefins contained in the 180° - 320°C fraction made by synthesising the same over Fe-Cu catalyst. The largest rate of conversion was achieved when the molar ration of C_nH_{2n} - H_2SO_4 = 1:2. Sulphonation experiments on various raw materials (Table 4) proved that the depth of conversion in one operation amounted to 73 - 81%. The remaining 19 - 27% of olefins can be used for a second sulphonation operation. Further experiments were carried out on the 180 - 320° fractions containing 32% olefins in order to separate the excess H_2SO_4 and re-use of the same in the cycle. According to the conclusions of A. Yu. Rabinovich and M. S. Il'in of the Moscow Branch of VNIIZh

The Preparation of Detergents From Olefins. SOV/65-58-8-10/16

the prepared detergents showed good surface-active properties. The most satisfactory results were obtained with solutions prepared from narrow fractions containing mostly C_{12} and C_{15} - C_{17} hydrocarbons and from the 230 - 320°C fraction. The detergent action of aqueous solutions can be further improved by the addition of carboxymethylcellulose. There are 4 Tables, 1 Figure and 10 References: 5 English, 1 French and 13 Soviet.

ASSOCIATION: VNII NP

1. Detergents--Preparation 2. Detergents--Materials
3. Ethylenes--Polymerization 4. Methanes--Fractionation

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SOV/65-58-12-3/16

AUTHORS: Rapoport, I. B.; Kruglikov, V. Ya. and Bol'shov I. I.

TITLE: The Development of a Highly Effective Synthesis Process From CO and H_2. (O razrabotke vysokoproizvoditel'nogo protsessa sinteza iz CO i H_2)

PERIODICAL: Khimiya i Tekhnologiya Topliv i Masel, 1958, Nr 12, pp 36 - 41 (USSR)

ABSTRACT: The following three problems have to be solved for achieving a highly effective industrial synthesis process on a stationary iron Fe catalyst: (1) heat elimination has to be at a required level while maintaining the necessary temperature for the synthesis process; (2) stable heat conditions have to be maintained and (3) the reaction $2CO = CO_2 + C$ has to be surpressed, and the negative effect of carbon deposition reduced to a minimum. The influence of circulation on the rate of the synthesis reaction has to be evaluated (Ref.1). The authors used the formula proposed by Zeligman and Anderson (Ref.2) and derived further equations. They found that under industrial conditions (when the concentration of H_2 equals 40 to 60%. and the rate of conversion equals 60to30%) an increase in the coefficient of circulation leads to a decrease in the reaction rate which is independent of

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The Development of a Highly Effective Synthesis Process from CO and H_2

the magnitude of the rate constant and consequently, also of the chemical properties of the catalyst. This decrease in the active component can be compensated by increasing the flow of mass of CO+ H_2 to the catalyst particle. An alternative way is to increase the reaction temperature which causes acceleration in the side reactions of methanisation and decomposition of CO. These assumptions were confirmed by experiments carried out on a pilot plant. The diameter of the reaction tube was 19, 21 and 23 mm. The height of the catalyst layer 4,000 mm; up to 2 litre of catalyst were used. Purified synthesis gas (CO : H_2 = 1 : 1.1 or CO : H_2 = 1 : 1.9), containing 15% of inert material, was used as starting material. Three different samples of catalysts were prepared for investigating the effect of circulation on the reaction rate when carrying out the process on catalysts having different macro-structures. The catalyst samples were preformed at pressures from 0 - 10,000 kg/cm² which caused changes in the density varying from 3.4 to 5.1 g/cm³ and changes in the porosity between 57 to 66%. Results are given in a Graph

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on page 39. The rate of conversion of CO + H_2O increases on granulated catalysts (when the preforming pressure equals 0 kg/cm^2) with increasing circulation coefficient. An increase in the circulation coefficient, when using catalysts preformed at a pressure of 10,000 kg/cm^2, leads to a decrease in the rate of conversion of CO+ H_2O. At 5,000 kg/cm^2 pressure the rate of conversion is practically independent of the circulation coefficient. These phenomena are explained. Results obtained during the investigation of the dependence of the degree of conversion on the circulation coefficient, and also on kinetic characteristics confirm the possibility of compensating the decrease in the conversion by changing the macrostructure of the catalyst. The most effective catalyst was obtained when preforming was carried out at a pressure of 5,000 kg/cm^2. Under the given synthesis conditions (volume rate equals 1,000 hours^{-1}, circulation coefficient equals 2 and the degree of conversion of CO+H_2 equals 70 - 80%) the decomposition of CO proceeds at a relatively fast rate, and in this way it was possible to achieve 20 - 30 day runs. Experiments were also carried out on decreasing the heat separation

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The Development of a Highly Effective Synthesis Process from CO and H_2

and on intensifying the heat elimination by decreasing the volume rate or the conversion rate. Runs were carried out at 70% degree of conversion and volume rates of 1000 & 400 hrs^{-1}, and it was found that at the latter volume rate the heat separation was reduced to 30%, and the length of the run increased from 33 to 90 days. The heat elimination can be intensified at a given height of a catalyst layer and circulation coefficient by decreasing the diameter of the reaction tubes. Reaction tubes with a diameter of 25.81 and 19 mm were tested at a height of the catalyst layer of 4,000 mm. and a circulation coefficient of 2. The process can be carried out in tubes with an internal diameter of 19 mm. The following optimum conditions for this process are specified: volume rate 1,000 hours^{-1}, pressure 30 atms, circulation coefficient 2, temperature 310°C, a ratio of CO:H_2 in the synthesis gas of 1:1.2; diameter of the reaction tube 19 mm and degree of conversion of CO + H_2 60%. Results of a 33-day run of the reactor, using an iron catalyst preformed at a pressure of 5,000 kg/cm^2, are given. Disadvantages of the

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The Development of a Highly Effective Synthesis Process from CO and H_2

process lie in the high yield of light products and the small diameter of the reaction tubes. It was also found that the yield of the middle fractions can be increased by using very active and selective catalysts and that larger diameter reaction tubes can be used when increasing the linear velocity of the gas. There are 1 Figure and 4 Soviet References.

ASSOCIATION: VNII NP

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5(4) SOV/20-123-5-33/59

AUTHORS: Rapoport, I. B., Kulakov, V. M.

TITLE: On the Problem of the Semiconductor Properties of Iron-Copper Catalysts of Synthesis and of the Activating Effect of an Alkaline Promotor (K voprosu o poluprovodnikovykh svoystvakh zhelezo-mednykh katalizatorov sinteza i aktiviruyushchem deystvii shchelochnogo promotora)

PERIODICAL: Doklady Akademii nauk SSSR, 1958, Vol 123, Nr 5, pp 887-890 (USSR)

ABSTRACT: According to the results of some previous papers (Refs 6, 7), there is a direct connection between the electrical conductivity and the activity of some catalysts (for example NiO). The authors assume that there must be a direct connection between the electric conductivity and the activity also in the case of a Fe-Cu-catalyst for the synthesis of oxygen-containing compounds and hydrocarbons from CO and H_2. In this case, the activating effect of the alkaline promoting agent must consist of the increase of the catalyst in electrical conductivity. The present paper deals with the results of the experimental investigations which were carried out to prove

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On the Problem of the Semiconductor Properties of Iron-Copper Catalysts of Synthesis and of the Activating Effect of an Alkaline Promotor

the above-mentioned assumptions. Carrying out of the experiments is discussed in short. Non-reduced Fe-Cu catalysts even at 230° have no noticeable electric conductivity and their resistance (for any temperature) was higher than 1,000,000 Ω. The resistance of the reduced catalyst samples, however, decreases with increasing temperature according to an exponential law. This dependence (which is characteristic of semiconductor materials) shows that reduced Fe-Cu catalysts for synthesis are typical semiconductors. Their activity increases with decreasing resistance and, therefore, with increasing electric conductivity. According to the results of this paper, there is a direct connection between the electric conductivity and the activity of iron copper catalysts in synthesis reactions. A diagram shows the influence of the potash content in the catalyst on its resistance and activity at 210°. The introduction of 0.5% potash increased the electric conductivity of the catalyst 1.5 times. A further increase in the K_2CO_3 content reduced the resistance and increased the activity of the catalyst. The promoting effect of potash (which is an acceptor impurity) can be explained by

SOV/20-123-5-33/50

On the Problem of the Semiconductor Properties of Iron-Copper Catalysts of Synthesis and of the Activating Effect of an Alkaline Promotor

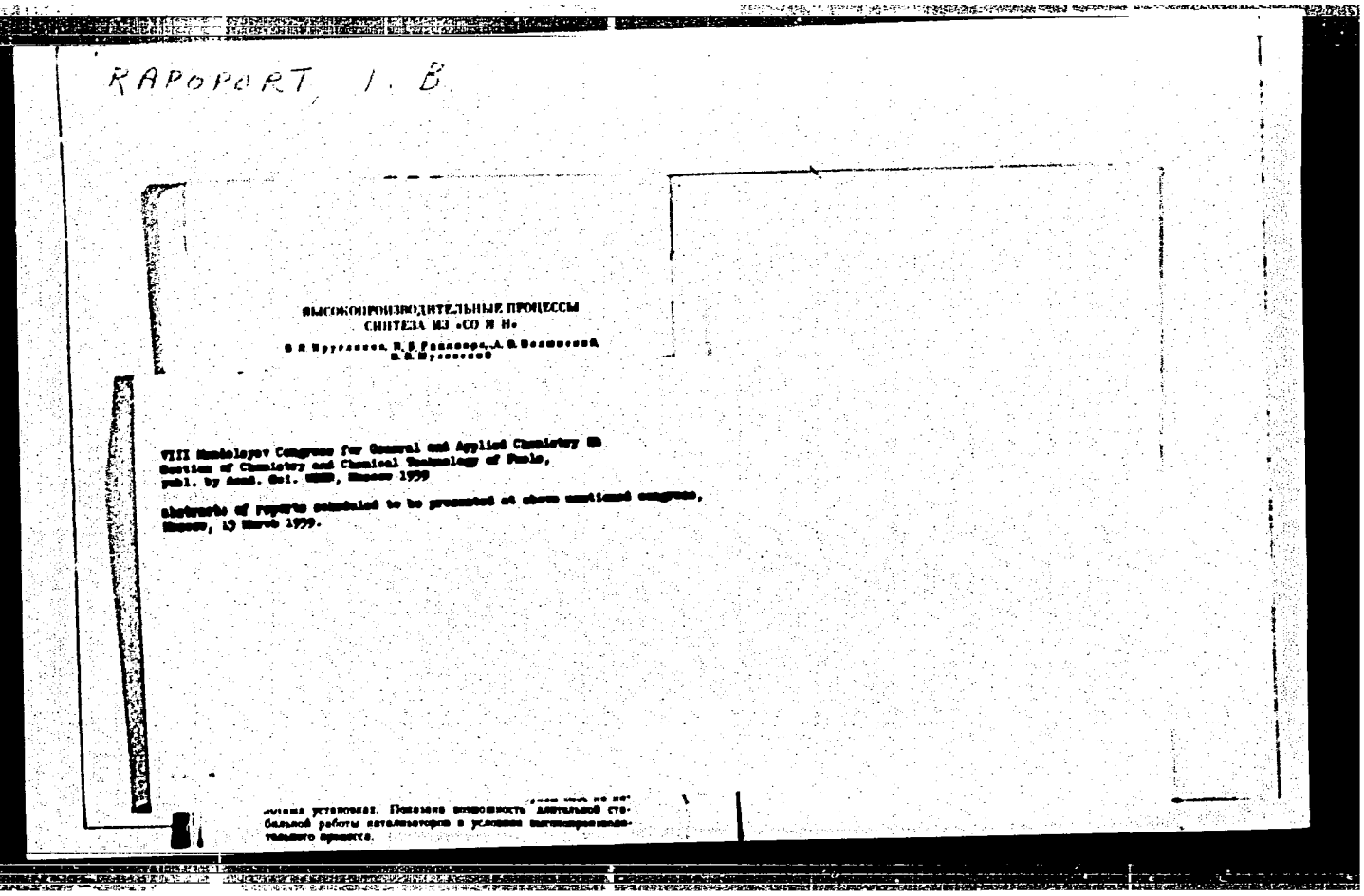
its influence upon the increase of the electric conductivity of the catalyst. There are 3 figures, 1 table, and 11 references, 7 of which are Soviet.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy institut po pererabotke nefti i gaza i polucheniyu iskusstvennogo zhidkogo topliva (All-Union Scientific Research Institute for Petroleum and Gas Refining and for the Production of Synthetic Liquid Fuel)

PRESENTED: July 17, 1958, by B. A. Kazanskiy, Academician

SUBMITTED: May 23, 1958

Card 3/3



RAPOPORT, I. B.

ВЫСОКОПРОИЗВОДИТЕЛЬНЫЕ ПРОЦЕССЫ
СИНТЕЗА ИЗ «СО И Н»

[illegible]

VIII Mendeleyev Congress for General and Applied Chemistry
Section of Chemistry and Chemical Technology of Fuels,
publ. by Acad. Sci. USSR, Moscow 1959

Abstracts of reports scheduled to be presented at above mentioned congress,
Moscow, 19 March 1959.

[illegible] устойчивая. Показана возможность длительной стабильной работы катализаторов в условиях пилотированного процесса.

RAPSPORT, I. B.

S/3400 77-1-1
SOV/63-4-6-15/17

AUTHORS: Meshkin, P. A., Kotenev, B. I., Veltzar'yeva, N. I., Sonkin, M. A., Kantser, V. I., Rapoport, I. B.

TITLE: Higher Aliphatic Alcohols From Solid Paraffin Oxidation Products

PERIODICAL: Khimicheskaya nauka i promyshlennost', 1959, Vol 4, Nr 6, pp 811-812 (USSR)

ABSTRACT: This is a summary of the article published in Khimiya i tekhnologiya topliv i masel, 1960, Nr 1, pp 24-27, see Abstract 77C-1.

ASSOCIATION: Scientific Research Institute for the Processing of Petroleum and Gas and for the Production of Synthetic Liquid Fuel (Nauchno-issledovatel'skiy institut po pererabotke nefti i gaza i polucheniyu iskusstvennogo zhidkogo topliva)

SUBMITTED: July 13, 1959
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3.

5(3) SOV/80-32-5-32/52

AUTHORS: Rapoport, I.B., Nefedov, B.K., Grakhova, S.G.

TITLE: On the Reaction of Dehydrogenation of Higher Paraffin Hydrocarbons Over Coal Catalysts

PERIODICAL: Zhurnal prikladnoy khimii, 1959, Vol 32, Nr 5, pp 1112-1121 (USSR)

ABSTRACT: The production of olefines from lower paraffin hydrocarbons is possible by means of dehydrogenation. The dehydrogenation of paraffin hydrocarbons with five and more carbon atoms is investigated here. At 450 - 510°C the dissociation of paraffin hydrocarbons takes place over activated coal with promotor. This reaction is accompanied also by dehydrogenation. The yield of liquid catalysate is 82 - 95%, the yield of gas 3 - 15%. The liquid products contained 20 - 30% unsaturated compounds. The raw material for the reaction was sintin, a product obtained from CO and H_2 over a Co-ThO_2-MgO catalyst. Promotors for the activated coal were salts of Na, Li, Rb, Cs and other metals. The promotors cause the increase of the H_2 : C_nH_{2n+2} ratio from 0.362 to 1.35. The best promotor is caustic soda followed by Na_2CO_3. Among the other metals a positive effect show only Li salts. The best carrier for the catalyst in the fraction 180 - 200°C is activ-

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On the Reaction of Dehydrogenation of Higher Paraffin Hydrocarbons Over Coal Catalysts

ated coal of type KAD. With the increase of the boiling point of the raw material the raction of dissociation plays an important role. The yield of liquid products decreases and coke and gas formation increases. Since at 500 - 510°C the dissociation reaction prevails, the temperature should be kept at 470 - 480°C. At a volume rate of 3 vol/vol · catalyst · hour the dehydrogenation reaction prevails. The catalyst KAD + 1% NaOH decreases its activity after 10 - 12 hours and must be regenerated by superheated steam for 10 hours. Experiments with the single hydrocarbon n-heptane have shown that a partial dehydrogenation takes place without dissociation and dehydrocyclization. There are 6 tables, 4 graphs and 4 references, 2 of which are Soviet and 2 American.

SUBMITTED: October 9, 1957

5(3) SOV/80-32-5-33/52

AUTHORS: Rapoport, I.B., Nefedov, B.K.

TITLE: On the Reaction of Dehydrogenation of Paraffin Hydrocarbons Over Chromium-Aluminum Catalysts

PERIODICAL: Zhurnal prikladnoy khimii, 1959, Vol 32, Nr 5, pp 1121-1125 (USSR)

ABSTRACT: The article is a continuation of [Ref 1] using a chromium-aluminum catalyst. Such a catalyst had been used by Z.I. Vozzhinskaya. Shuykin had converted n-pentane to pentene with a yield of 31% using a catalyst with the ratio Al_2O_3 : Cr_2O_3 : K_2O = 90.7 : 5.6 : 3.7 in mole %. The sintin fractions 180 - 200°C and 300 - 380°C were used as raw material. The principal reaction of the first fraction is dehydrogenation. The hydrogen content in the gas reaches 65%. In the second fraction dissociation is also observed. The best catalyst for the dehydrogenation of this fraction contains 8% Cr_2O_3, whereas for the 180 - 200°C fraction the content should be 16%. The catalysts

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On the Reaction of Dehydrogenation of Paraffin Hydrocarbons Over Chromium-Aluminum Catalysts

are active for 28 hours and are regenerated in a stream of hot air at 600 - 650°C.
There are 5 tables and 3 references, 2 of which are Soviet and 1 German.

SUBMITTED: October 9, 1957

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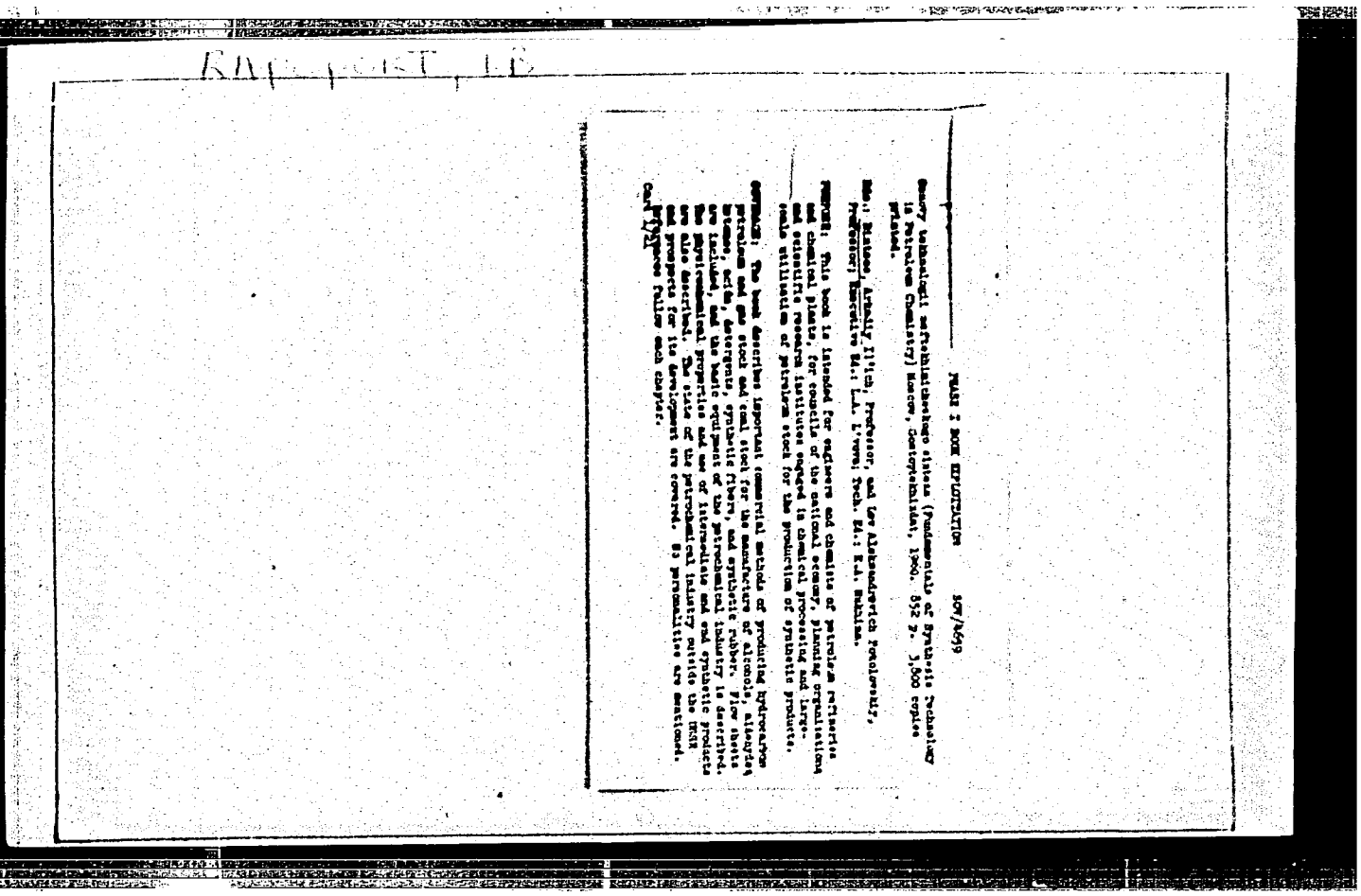

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USSR / BOOK EXPLOITATION

Nauchno-tekhnicheskiy sovet po khimicheskoy tekhnologii (Scientific Technological Council for Chemical Technology)

Nauka v Petrolevoy Khimii (Progress of Science in Petroleum Chemistry) Moscow, Gostoptekhizdat, 1960. 352 p. 3,500 copies printed.

Ed.: Platonov, Arkadiy Il'ich, Professor, and Ger Aleksandrovich Tsvetkovskiy, Professor; Executive Ed.: L.A. Ivanova; Tech. Ed.: E.A. Maksina.

PURPOSE: This book is intended for engineers and chemists of petroleum refineries and chemical plants, for councils of the national economy, planning organizations and scientific research institutes engaged in chemical processing and large-scale utilization of petroleum stock for the production of synthetic products.

COVERAGE: The book describes important commercial methods of producing hydrocarbon petroleum and gas stock and coal stock for the manufacture of alcohols, alkylated benzene, acids, detergents, synthetic fibers, and synthetic rubber. Flow sheets of the equipment of the petrochemical industry is described. Its included, and the basic properties and use of intermediate and end synthetic products and petrochemical properties and use of intermediate and end synthetic products are also described. The state of the petrochemical industry outside the USSR and prospects for its development are covered. 85 personalities are mentioned.

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VELIZAR'YEVA, N.I.; RAPOPORT, I.B.; MAN'KOVSKAYA, N.K.; BARSEGYAN, I.B.; SHIMAN, A.M.; BABAYEV, V.I.; SUKHOTERIN, I.S.

Industrial experience in the oxidation of paraffins from sulfur-bearing crudes. Khim.i tekh.topl.i masel 5 no.7:11-16 Jl '60.

1. Vsesoyuznyy nauchno-issledovatel'skiy institut po pererabotke nefti i gazov i polucheniyu iskusstvennogo zhidkogo topliva, NII SZhIMS i Shebekinskiy kombinat sinteticheskikh zhirnykh kislot i zhirnykh spirtov.

(Paraffins) (Oxication)

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1.1800 1521 1087

S/081/61/000/020/044/089
B107/B101

AUTHORS: Polukarov, M. N., Geraseva, S. S., Rapoport, I. P.

TITLE: Effect of mercury chloride additions to electrolytes on the absorption of hydrogen by steel during cathodic polarization

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 20, 1961, 258, abstract 20I137 (Izv. Yestestvennonauchn. in-ta pri Permsk. un-te, v. 14, no. 4, 1960, 3 - 11)

TEXT: The authors found the following: Addition of $HgCl_2$ to NaOH solutions considerably reduces the tensile strength limit of steel subjected to cathodic polarization in these solutions. Such an effect is not observed during polarization in H_2SO_4 solutions with the same addition. The tensile strength also decreases considerably during zinc-plating of steel wire in dilute cyanide and zincate electrolytes. This is not observed during zinc-plating in acid solutions. The changes in tensile strength of steel and the differences of these changes in the polarization in alkaline and acid electrolytes are explained by the different

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