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# Science & Technology

## USSR: Chemistry

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**Distinctive Features of the Critical Conditions of the Ignition of Monosilane With Oxygen in the Low-Pressure Range**

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[Article by V.V. Azatyan, A.A. Vartanyan, V.A. Kalkanov, and A.A. Shavard, Structural Macrokinetics Institute, USSR Academy of Sciences, Chernogolovka]

UDC 541.126.2:[546.281+546.21]+621.43.019.2

[Abstract] The authors of the study reported herein examined the distinctions of the critical conditions of the ignition of monosilane ( $\text{SiH}_4$ ) with  $\text{O}_2$  at low pressures. Specifically, they sought to clarify the possible participation of excited states in the branching of chains and to determine the dependence of the second ignition threshold on the surface state and its properties. The authors used a static vacuum unit to perform a series of experiments examining the self-ignition of  $\text{SiH}_4$  with oxygen in the presence of various diluents (Ar, He,  $\text{CO}_2$ ,  $\text{H}_2$ ,  $\text{N}_2$ , and  $\text{SiH}_4$ ). The experiments established that unlike He, Ar, and  $\text{H}_2$ , the additive  $\text{N}_2$  greatly (by 20%) increases the pressure of  $\text{SiH}_4$  and  $\text{O}_2$  at the first ignition threshold. The temperature dependence of the first threshold is shown to weaken as the content of  $\text{SiH}_4$  in the mixture is increased. The shape of the ignition region's boundaries is discovered to be "anomalous." Both the first and second self-ignition thresholds are found to be state dependent. Measurements of the temperature dependence of the first thresholds of mixtures with different silane contents show that both the effective activation energy of the threshold and the critical pressure depend on the mixture's composition. In other words, at different pressure intervals corresponding to different silane contents, changes occur in the competing reactions determining the first threshold. That is to say, the totality of the process' main stages are essentially altered. Figures 3; references: 12 Russian, 3 Western.

**Oxidizing Condensation of Methane With the Participation of  $\text{O}_2$  and  $\text{N}_2\text{O}$  in the Absence of Catalysts**

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in Russian Vol 32 No 2, Mar-Apr 91 (manuscript  
received 18 Jan 90) pp 260-266

[Article by N.I. Ilchenko, L.N. Rayevskaya, Yu.I. Shmyrko, and V.M. Vorotyntsev, Physical Chemistry Institute imeni L.V. Pisarzhevskiy, UkSSR Academy of Sciences, Kiev]

UDC 541.124:542.943'953:[547.211+546.21+546.172.5]

[Abstract] The oxidizing condensation of methane with the participation of oxygen in the presence of metal oxides or metal-like catalysts is a heterogeneous-homogeneous process that includes the heterogeneous

generation of methyl radicals and homogeneous stages of their further transformations. The same process can also occur in the absence of catalysts. In view of this fact, the authors of the study reported herein examined the reaction of  $\text{CH}_4$  and  $\text{O}_2$  at atmospheric pressure under the same temperature and concentration conditions at which catalytic condensation occurs. Because heterogeneous catalysts are also capable of accelerating the oxidizing condensation of methane with the participation of  $\text{N}_2\text{O}$ , they also examined the gas-phase reaction of  $\text{CH}_4$  and  $\text{N}_2\text{O}$ . The experiments were conducted in a flowthrough differential reactor at atmospheric pressure and at temperatures between 500 and 750°C. The partial pressure of the methane was varied between 20 and 40 kPa, and the partial pressure of the oxygen was varied between 6 and 14 kPa. Helium was used as a diluent gas. The total flow rate of the gas mixture was varied between 30 and 150  $\text{cm}^3/\text{min}$ . It was discovered that the reaction of methane with  $\text{O}_2$  or  $\text{N}_2\text{O}$  in the absence of a catalyst at about 700° at atmospheric pressure results in the formation of oxidizing condensation products (ethane, ethylene, propane, propylene) and carbon oxides. In the case of a reaction with  $\text{O}_2$  the total transformation occurs more quickly, but the product selectivity of oxidizing condensation is lower than when  $\text{N}_2\text{O}$  is used as an oxidizing agent. The tests also demonstrated that filling the free space in the reactor with quartz glass decreases the total speed of the process sharply while, at the same time, increasing ethane selectivity. The authors propose a diagram of the reaction and discuss its kinetics through a series of equations. Figures 3, tables 2; references 16: 11 Russian, 5 Western.

**Pyrolysis of Methane Under Static Conditions in the Temperature Range From 1,100 to 1,400 K**

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received 20 Feb 90) pp 267-273

[Article by V.S. Arutyunov, V.I. Vedeneyev, R.I. Moshkina, and V.A. Ushakov, Chemical Physics Institute imeni N.N. Semanova, USSR Academy of Sciences, Moscow]

UDC 541.127:542.921.4:547.211

[Abstract] Pyrolysis processes are conducted over a very broad temperature ranged depending on the purpose of the individual process. Pyrolysis processes may, for example, occur at temperatures below 1,000 K, between 1,000 and 1,800 K, or above 1,800 K. A relatively new area of using natural gas, i.e., the methane reduction of  $\text{SO}_2$  in the off gases in nonferrous metallurgy at temperatures from 1,200 to 1,500, has necessitated research on methane pyrolysis under static conditions in the temperature range selected by the authors of the study reported herein. They conducted their pyrolysis experiments under static conditions in quartz reactors 100 mm long

and 46 and 21 mm in diameters. The reaction mixture was transferred into a pre-evacuated reactor. The cold space in the reactor ( $d = 46$  mm) amounted to  $\leq 0.2\%$  of the total space. After a specified amount of time, the reaction mixture was released into a sampling column and subjected to chromatographic analysis to determine the mixture's content of  $\text{CH}_4$ ,  $\text{H}_2$ ,  $\text{N}_2$ ,  $\text{O}_2$ ,  $\text{CO}$ , and  $\text{C}_2$  hydrocarbons (ethane, ethylene, and acetylene). The precision of determining the content of the various gases in the mixture was about 10%. The methane used contained about 1.5% air as its principal impurity and did not contain any recordable amounts of  $\text{C}_2$  hydrocarbons. Distillation in liquid nitrogen was used to reduce the average air content to 0.5% and the average oxygen content to 0.1%. The authors proceeded to compute monomolecular reaction rate constants and to obtain data about the effect that pressure, reactor surface, and hydrogen and oxygen impurities exert on the process. They then proceed to derive an express for the monomolecular rate constant of methane pyrolysis that well describes not only their own data but also that obtained by a series of other authors for the 1,100 to 1,700 K temperature range as well. This expression is as follows:  $R = (3.0 \pm 1.0) \times 10^{12} \exp[-343,000 \pm 12,000/RT]$  (in inverse seconds). Figures 5, tables 4; references 24: 5 Russian, 19 Western.

#### Liquid-Phase Oxidation of Propylene on Applied Palladium Mono- and Bimetallic Catalysts in Acetic Acid

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[Article by A.V. Karandin, Ye.V. Gusevskaya, V.A. Likholobov, A.I. Boronin, and E.M. Moroz, Catalysis Institute, Siberian Department, USSR Academy of Sciences]

#### UDC

541.128.13:541.124-145.15:542.943.7:546.98:547.313.3

[Abstract] The authors of the study reported herein examined the properties of palladium mono- and bimetallic catalysts in the liquid-phase oxidation of propylene to propylene glycol and its acetates in acetic acid solutions of nitric acid in the presence of heterogeneous catalysts. The said catalysts were prepared by applying  $\text{PdCl}_2$ ,  $\text{H}_2\text{PtCl}_2$ ,  $\text{RhCl}_6$ , or their binary mixtures onto various carriers and then reducing the adsorbed complexes by means of sodium nitrate in an alkaline medium. The following were used as carriers: the carbon material Siburnit (specific surface,  $600 \text{ m}^2/\text{g}$ ),  $\text{SiO}_2$  (specific surface,  $207 \text{ m}^2/\text{g}$ ), and  $\text{TiO}_2$  (specific surface,  $110 \text{ m}^2/\text{g}$ ). The carriers' particle sizes ranged from 15 to 90  $\mu\text{m}$ . The catalysts were tested on a unit with a vibrating thermostatted reactor equipped with a reverse cooler and sampler. The tests were conducted in a static mode or with a gas mixture flowing through. NSR spectroscopy, gas-liquid chromatography, and spectrophotometry studies were performed on the test specimens. It was

discovered that solid catalyst is involved in the main part of the reaction that occurs, although a portion of the reaction occurs in a solution as a result of the washing away of active components from the carrier. The palladium-platinum catalysts were found to be the most resistant to deactivation due to the reaction medium. The studies performed also established the formation of cyanide metal complexes on the carrier surface as one of the causes of catalyst deactivation. Adding a second method to the catalyst composition, which results in modification of the properties of the heterogeneous contact and increasing the catalyst's resistance to the effect of the reaction medium and to deactivation processes, was suggested as a possible way of regenerating the catalysts. Figures 2, tables 3; references 16: 5 Russian, 10 Western.

#### A Study of the Mechanism of Olefin Metathesis and the Process of the Formation of Active Centers on Photoreduced Molybdenum Silicate Catalysts. 5. Metathesis of Esters of Unsaturated Fatty Acids

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[Article by M.Yu. Berezin, V.M. Ignatov, P.S. Belov, I.V. Yevlev, B.N. Shelimov, and V.B. Kazanskiy, Moscow Oil and Gas Institute imeni I.M. Gubkin and Organic Chemistry Institute imeni N.D. Zelinskiy, USSR Academy of Sciences, Moscow]

UDC 541.128:541.145:546.77'284:547.262'39:542.95

[Abstract] In a continuation of their previously published studies on the mechanism of olefin metathesis and the process of the formation of active centers on photoreduced molybdenum silicate catalysts, the authors of the study reported herein examined the metathesis of esters of fatty acids. Type KSK-2.5 silica gel was used as the carrier, and ethyl ethers of three different plant oils (olive, sunflower, and flax) were used as a raw material for preparing the ethyl ethers. An ethyl ether mixture was prepared by interesterification of triglycerides of the oils with ethyl alcohol.  $\alpha$ -Olefins (hexene-1, heptene-1, nonene-1, and decene-1) and symmetrical olefins (decene-5, dodecene-6, tetradecene-7, and octadecene-9) obtained by metathesis of the respective  $\alpha$ -olefins were used as comethesizing agents. n-Heptene freshly distilled over metallic sodium was used as a solvent. The esters were metathesized and comethesized in a glass reactor equipped with a thermostating sleeve, magnetic stirrer, and sampling tube. Liquid-phase samples were taken periodically and subjected to gas chromatographic analysis. It was discovered that molybdenum silicate catalyst that has been photoreduced in CO and processed with cyclopropane possesses a high activity and productivity

in reactions of the metathesis of esters of unsaturated fatty acids (including ethyl oleate, ethyl linoleate, and ethyl linoleate) and the cometathesis of ethyl oleate with decene-5 and other olefins. The activation energy of this reaction was estimated at 26.8 kJ/mol in the Arrhenius coordinates  $\ln W_0 - 1/T$ . The products of the cometathesis with decene-5 of ethyl ethers of acids having different degrees of unsaturation with decene-5 and that were produced on the basis of the three aforesaid plant oils were analyzed. Finally, the activity and productivity of the photoreduced catalyst molybdenum silicate catalyst reported herein were compared with those of other known catalysts. The Mo/SiO<sub>2</sub> catalyst described by the authors was determined to be notably superior its known analogues on both counts. The new catalyst is further distinguished by the fact that unlike its known counterparts, it does not necessitate the use of toxic cocatalysts. Figures 3, tables 5; references 20: 5 Russian, 15 Western.

#### The Kinetics and Mechanism of the Aromatization of n-Hexane on Aluminoplatinum Catalyst

917M0167F Moscow KINETIKA I KATALIZ  
in Russian Vol 32 No 2, Mar-Apr 91 (manuscript  
received 22 Aug 89) pp 390-394

[Article by M.S. Kharson, T.R. Dzigvashvili, A.V. Dolidze, and S.L. Kiperman, Organic Chemistry Institute imeni N.D. Zelinskiy, USSR Academy of Sciences, Moscow, and Physical and Organic Chemistry Institute imeni P.I. Melikishvili, GSSR Academy of Sciences, Tbilisi]

UDC 541.128.13:541.124:546.92'623:547.216:547.526

[Abstract] In an attempt to develop a kinetic model of the process of the aromatization of n-hexane on a commercial aluminoplatinum catalyst, the authors of the study reported herein examined the aromatization of n-hexane in the presence of the industrial aluminoplatinum catalyst IP-62 (0.5% Pt) in the range of temperatures from 370 to 420° in a glass flowthrough-circulation reactor. The ongoing partial pressures of n-hexane, hydrogen, and isohexane were varied within the respective ranges of 1 to 60, 520 to 980, and 0.56 to 20 GPa, and the space velocities were varied in the interval from 3 to 150 hours. Catalyst was loaded into the reactor in amounts ranging from 1 to 5 g, and the degree of transformation into aromatic compounds ranged from 0.07 to 0.34. Kinetic isotope effects were measured as the ratio of the reaction rates in hydrogen and deuterium atmospheres given identical degrees of n-hexane transformation, i.e.,  $\beta = r_H/r_D$ . Preliminary experiments entailing varying the catalyst grain sizes and circulation rates demonstrated that under the given conditions on grains 0.1 to 0.25 mm in size, the reaction occurs in the kinetic range. In the temperature range studied, the reaction products were also observed to contain light hydrocarbons formed during hydrogenolysis (<4%) and linear olefins. Methylcyclopentane and methylcyclopentene were also likely formed but could not be isolated

chromatographically. The isomerization products had a close-to-equilibrium makeup. The absence of any inhibition of the reaction by the hydrogenolysis products was confirmed experimentally. Benzene and toluene added in amounts that were triple or quadruple the ongoing benzene concentrations in the cycle did not affect the aromatization rate either. The data obtained were used to plot conversion curves and to develop a schematic of the process of aromatization of n-hexane that included eight stages and two routes. Figures 3, table 1; references 18: 11 Russian, 7 Western.

#### The Spectral and Kinetic Characteristics of the Photoactivated Adsorption of Oxygen on $\gamma$ -Al<sub>2</sub>O<sub>3</sub>

917M0166G Moscow KINETIKA I KATALIZ in Russian  
Vol 32 No 2, Mar-Apr 91 (manuscript received  
6 Feb 90) pp 428-432

[Article by A.O. Klimovskiy and A.A. Lisachenko, Physics Scientific Research Institute and Leningrad State University]

UDC 541.145:541.183.2:[546.21+546.623]:543.51

[Abstract] Because aluminum oxide is capable of affecting the equilibrium of photochemical reactions in the upper layers of the atmosphere (specifically, the photoactivated decomposition of ozone), the authors of the study reported herein decided to study photoactivated reactions of Al<sub>2</sub>O<sub>3</sub> with O<sub>2</sub>. Specifically, they examined the nature of photoactivated centers of the adsorption and desorption of O<sub>2</sub> on Al<sub>2</sub>O<sub>3</sub> and to determine their kinetic and spectral characteristics. In essence, the method entailed creating a gas flow at a dynamic pressure of 10<sup>-6</sup> to 10<sup>-3</sup> mm Hg over the study adsorbent and illuminating the specimen with monochromatic radiation at wavelengths from 200 and 600 nm inclusively. The gas flow leaving the reactor with the specimen was continuously monitored with a mass spectrometer, thus making it possible to establish the change in its composition and quantity. As test specimens the authors used weighted 50-mg portions of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> with a specific surface of 200 m<sup>2</sup>/g. The specimens contained no more than 10<sup>-3</sup>% Fe, Cr, and Mg impurities and no more than 10<sup>-2</sup>% silicon and were purified by heating to 1,000 K directly in the reactor in an O<sub>2</sub> flow at 5 x 10<sup>-2</sup> mm Hg for 30 hours. The experiments demonstrated that irradiating  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> with light under the specified conditions causes a shift in the adsorption-desorption equilibrium. The authors succeeded in identifying a photoabsorption with an excitation spectrum that coincided with the absorption of F<sup>+</sup> centers and a photodesorption that was maximally effective in the case of excitation at a wavelength of about 300 nm. The quantum yields of the processes and cross section of O<sub>2</sub> capture by a photoactivated center were determined. The latter amounted to between 3 and 5 x 10<sup>-18</sup> cm<sup>2</sup>, which is close to the cross section for ZnO but much less than the corresponding value for BeO. Figures 2; references: 8 Russian, 6 Western.

**Phase Transformations in the Structure of Iron-Containing Catalysts of the Process of Direct Oxidation of Hydrogen Sulfide Into Sulfur Caused by a Thermal Radiation Effect**

917M0167H Moscow KINETIKA I KATALIZ in Russian Vol 32 No 2, Mar-Apr 91 (manuscript received 25 Jul 89) pp 433-438

[Article by A.Yu. Adzhiyev, B.Ya. Adigamov, V.V. Lunin, I.I. Miroshnichenko, D.M. Panteleyev, P.V. Ryabchenko, M.V. Sadovnichaya, Yu.I. Solovestkiy, and Yu.V. Taletskiy, Belgorod Construction Materials Technology Institute imeni I.A. Grishmanov, Chemistry Department of Moscow State University imeni M.V. Lomonosov, and All-Union Gas Refining Scientific Research and Design Institute, Krasnodar]

UDC

541.128.13:546.76'72'46-44:539.172.2:546.02:542.943.7:546.221.543.422

[Abstract] The authors of the study reported herein examined the use of high-power beams of accelerated electrons with energies up to 2.0 MeV in treating Zn-Cr-Fe catalysts for direct oxidation of hydrogen sulfide into sulfur. For their studies, they used a Zn-Cr-Fe-based catalyst developed by the AzINEFTEKhIM in Baku. The tests were conducted for 700 hours and used hydrogen sulfide-containing gas that consisted mainly of CO<sub>2</sub> but also contained the following (%): H<sub>2</sub>S, 3 to 6; N<sub>2</sub>, 2; CH<sub>4</sub>, 0.5; C<sub>2</sub>H<sub>6</sub>, 0.3; C<sub>3</sub>H<sub>8</sub>, 1.1; and C<sub>4</sub>H<sub>10</sub>, 0.2. The temperature was kept between 540 and 570 K, and the O<sub>2</sub>:H<sub>2</sub> ratio was varied from 0.5 to 0.75, with the contact time amounting to 0.72 seconds. A flowthrough microreactor was used to determine catalytic activity in the reaction. The resultant hydrogen sulfide-containing gas, which consisted mainly of CO<sub>2</sub>, included 3% H<sub>2</sub>S. Air was used as an oxidizing agent, and the O<sub>2</sub>:H<sub>2</sub>S ratio was varied from 0.5 to 1. An ELV-2 commercial electron accelerator was used for the thermal irradiation of the test catalysts. The Zn-Cr-Fe catalysts tested proved to permit the formation of active spinel structures with a minimum irradiation time. Treatment of coked catalytic systems resulted in the effective removal of coke products and sulfur-containing reaction products. Catalysts used for direct oxidation of hydrogen sulfide into sulfur for a protracted period were observed to undergo a reversible phase transition associated with the formation of iron sulfide structures. Figures 4; references 11 : 10 Russian, 1 Western.

**Carriers for Catalysts of Organic Synthesis. 2. The Pore Structure of Aluminum Oxides Produced by Heat Treatment of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>**

917M0167I Moscow KINETIKA I KATALIZ in Russian Vol 32 No 2, Mar-Apr 91 (manuscript received 1 Feb 90) pp 447-454

[Article by B.M. Fedorov, V.Ya. Danyushevskiy, V.L. Balashov, and A.S. Berenblyum, All-Union Scientific Research Institute of Organic Synthesis, Moscow]

UDC 541.128.35:546.623-31:542.46:539.26+62-405.8

[Abstract] The authors of the study reported herein studied the genesis of the pore structure of pseudo-boehmite-type  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> subjected to controlled heat treatment. A-64K commercial  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> produced by the Ryazan Oil Refinery was roasted at temperatures of 550 to 1,000° for 1 to 8 hours. It was discovered that sintering conducted under the said condition is coalescent in nature. The specimens maintain their monodisperse structure, but the predominant pore radius increases from 80 to 125 angstroms. The sharpest change in pore structure occurs between 1,000 and 1,200° and is associated with the formation of an  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> phase. A new system of wider pores forms, and specimens containing between 40 and 70%  $\alpha$ -phase have a bimodal curve of pore distribution by radius with maxima at 125 and 480-600 angstroms. The formation of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> results in the formation of a new monodisperse structure that undergoes coalescent sintering between 1,200 and 1,350°. This second sintering in turn results in an even increase in pore size to 1,000 angstroms. Each of these heat treatment-induced changes in pore structure is discussed in detail, and the data are recommended for use in selecting methods of synthesizing aluminum oxide carriers having a complex phase composition so that they will have the required pore structure. Figures 3, table 1; references 22: 20 Russian, 2 Western.

**An Investigation of Catalysts and Catalytic Combustion Reactions. 10. Distinctive Features of Creating Strong Carriers for Fluidized Bed Catalysts by Using Liquid Formation**

917M0167J Moscow KINETIKA I KATALIZ in Russian Vol 32 No 2, Mar-Apr 91 (manuscript received 26 Sep 89) pp 455-460

[Article by M.N. Shepeleva, Z.R. Ismagilov, R.A. Shkrabina, and I.A. Ovsyannikova, Catalysis Institute, Siberian Department, USSR Academy of Sciences, Novosibirsk]

UDC

541.128.13:541.128.35:546.623:541.18.02:539.4.01

[Abstract] The authors of the study reported herein examined the possibility of producing strong granules of Al<sub>2</sub>O<sub>3</sub> with a rather narrow range of mechanical indicators that are suitable for use as carriers of catalysts for catalytic heat generators. They also set out to find ways of reducing the amount of weak granules produced. Aluminum oxide was formed into spherical granules by the liquid method based on successive treatments with a peptizer and coagulant. The granules' average crushing strength ( $P_{av}$ ) was determined on the basis of a sample of 30 granules, the minimal crushing strength ( $P_{min}$ ) was determined from the five lowest readings, and  $P_{max}$  was determined analogously. Aluminum hydroxide with a pseudo-boehmite structure, moisture content of 78% (by weight), and primary particle size of 8-10 nm was used (this specimen was labeled AH-1). To further increase

the strength of the end product, some of the AH-1 was subjected to successive peptizing and coagulation and subsequently labeled AH-2. The two specimen types were then subjected to a series of tests to determine their structural and mechanical characteristics. The authors concluded that the creation of carriers and catalysts capable of not breaking down under the effects of chemical, thermal, and dynamic loads arising in fluidized bed equipment should proceed along the route of finding the conditions required to produce granules of uniformly high strength and to reduce the number of weak granules in specimens. Familiar technological techniques that make it possible to reduce the spread of specimens' mechanical indicators are recommended for use with aluminum hydroxides of the AH-1 type. These methods are shown to be unacceptable for use with aluminum hydroxides of the AH-2 type. The maximum possible strength values for actual defect structures of both types are estimated. Figures 2, tables 2; references 10: 9 Russian, 1 Western.

#### **Oxidation With Pd (II) Complexes in the Presence of Cu (II) in Aqueous Solutions**

917M0167K Moscow KINETIKA I KATALIZ  
in Russian Vol 32 No 2, Mar-Apr 91 (manuscript  
received 19 Jan 90) pp 477-480

[Article by L.V. Kashnikova, Organic Catalysis and Electrochemistry Institute imeni D.V. Sokolskiy, KaSSR Academy of Sciences, Alma-Ata]

UDC 541.128:541.124-145.2:546.262.3-  
31:546.98'562'131:541.127

[Abstract] The authors of the study reported herein examined the kinetics of the reaction of the oxidation of low-concentration (<1%) CO by Pd (II) and Cu (I, II) complexes in the presence of oxygen and without it. Their primary objective in so doing was to determine whether this reaction can be used in processes of scrubbing carbon oxide from gases. The studies were conducted in a flowthrough, long-necked flask-type thermostatted reactor with a drop sampler that created kinetic conditions. The kinetics of the oxidation of low-concentration CO by Cu (II) and Pd (II) complexes in aqueous solutions was found to differ from the laws governing high-concentration CO: Oxygen does not exert a modifying effect, the reaction with respect to Pd (II) has an order of less than 1, the reaction with respect to CO had an order of 1, and the reaction's speed decreases as the concentration of chloride ions increases. Cu (I, II)

chlorides sharply reduce the speed of oxidation of CO by Pd (II). Consequently, aqueous solutions of Pd (II) halide can only be used to scrub highly concentrated CO from low-concentration gases in the absence of excesses of chloride ions and copper salts. Figures 3; references 10: 9 Russian, 1 Western.

#### **The Effect of the Nature of Surface Centers on Forms of Methanol Adsorption on Cr<sub>2</sub>O<sub>3</sub> and Cr-Mo-O Catalysts**

917M0167L Moscow KINETIKA I KATALIZ  
in Russian Vol 32 No 2, Mar-Apr 91 (manuscript  
received 11 Dec 89) pp 486-489

[Article by N.A. Osipova, A.A. Davydov, and L.N. Kurina, Tomsk State University imeni V.V. Kuybyshev and Catalysis Institute, Siberian Department, USSR Academy of Sciences]

UDC 541.128.3:541.183.2:547.261:546.77'76-  
31:543:422.4

[Abstract] The authors of the study reported herein examined the differences in the mechanisms of the formation of surface methoxyl compounds when methanol is adsorbed on total- and partial-oxidation catalysts (Cr<sub>2</sub>O<sub>3</sub> and Cr-Mo-O catalysts, respectively). IR spectroscopy was used to study the adsorption of methanol with preadsorption of NH<sub>3</sub>, CO, and H<sub>2</sub>O on the surfaces of the two aforementioned catalysts. Even at ambient temperature, the reaction of methanol with Cr<sub>2</sub>O<sub>3</sub> results in the formation of surface methoxyl structures that are spectrally identified from the following adsorption bands: 1,090 (ν CO), 2,840, 2,940 (ν<sub>s,as</sub> CH), and 1,470 cm<sup>-1</sup> (δ CH). The surface methoxyl compounds formed when methane is adsorbed on a mixed Cr-Mo-O catalyst are identified on the basis of the following adsorption bands: 1,080 (ν CO), 1,380, 1,470 (δ CH), 2,865, and 2,970 cm<sup>-1</sup> (ν<sub>s,as</sub> CH). Thus, on chromium oxide alcohol is activated on the basis of a heterolytic mechanism involving the participation of nucleophilic oxygen of the surface and resulting in the formation of CH<sub>3</sub>O<sup>-</sup> particles stabilized on a Lewis acid center. The formation of surface methoxyl structures on a Cr-Mo-O catalyst, on the other hand, involves mobile surface protons. In conclusion, the authors note a definite similarity in the effect mechanism of homogeneous and heterogeneous catalysts of alcohol oxidation in carbonyl compounds. They assert that this commonality will make it possible to find new approaches to controlling the process' selectivity by regulating the function of the surface's active centers. Figures 3; references 8: 5 Russian, 3 Western.

### The Problem of the Thermal Stability of Catalytic Fuel Combustion Catalysts

917M0159A Kiev *KHIMICHESKAYA  
TEKHNOLOGIYA* in Russian No 4, Jul-Aug 91  
(manuscript received 22 Feb 91) pp 12-17

[Article by O.A. Kirichenko, A.O. Bobyрева, R.A. Shkrabina, L.F. Melgunova, and N.A. Koryabkina, Catalysis Institute, Siberian Department, USSR Academy of Sciences, Novosibirsk]

UDC 66.097.373:620.17

[Abstract] Thermomechanical stability, i.e., preservation of high strength indicators after heat treatment at temperatures ranging from 600 to 1,200°C, is one of the key indicators considered when developing catalysts for fuel combustion in a fluidized bed. The authors of the study reported herein studied the thermal stability of aluminum oxide carriers and catalysts used for fuel combustion. Specifically, they studied  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and two catalysts based on them, i.e., the AlCuCr catalyst IK-12-70 and the AlMnCr catalyst IK-12-72. All were subjected to heat treatment under various conditions simulating different versions of actual processes. The crushing strength of the granules (P) was determined as the arithmetic mean of 30 strength values of single granules on an MP-9S instrument. The extremal values were computed from 5 minimal (P<sub>min</sub>) and 5 maximal (P<sub>max</sub>) strength values. An MP-14D instrument was used to determine the granules' strength under dynamic conditions (fatigue strength). The effect of moisture and temperature difference on granules in catalytic fuel combustion devices was also studied. The studies indicated that thermal stability was very much dependent on cooling conditions, granule heating temperature, and fraction composition of the specimens. It was discovered that cooling the granules in a manner that entailed complete blocking of their pores resulted in a much greater reduction in mechanical strength than did cooling by vapor and rapid evaporation of moisture from the pore space. The level of strength following thermal stability testing was also found to differ greatly depending on the nature of the specimen. The  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>-based IK-12-72 catalysts remained the strongest after testing. The experiments also revealed that when sewage is burned, the feed of the sewage into the fluidized bed should be set up in the form of steam-gas or else it should be fed into the lower-temperature areas above the fuel combustion zone by using a nonisothermal-type device. The allowable temperature for the sewage input zone should be determined on a case-by-case basis by using a testing procedure described by the authors. The tests described will make it possible to differentiate carriers and catalysts of different natures and may also be recommended as a test for use when developing catalysts to burn sewage and activated sludge. Figures 3, tables 3; references 10 (Russian).

### The Hydration of Acetonitrile on a Copper Catalyst in the Presence of Salt Additives

917M0159B Kiev *KHIMICHESKAYA  
TEKHNOLOGIYA* in Russian No 4, Jul-Aug 91  
(manuscript received 25 Mar 91) pp 24-27

[Article by T.M. Mokrivskiy and I.O. Mikhaylishin, Lvov Polytechnic Institute]

UDC 547.398.1:66.061.5

[Abstract] The authors of the study reported herein studied the effect of copper, aluminum, and chromium sulfates on the hydration rate of acetonitrile on a copper catalyst prepared by the reduction of copper oxide by hydrogen. Copper catalyst with a particle size of 0.05 to 0.1 mm and a specific surface of  $15 \times 10^3$  m<sup>2</sup>/kg was produced by breaking copper carbonate down into copper oxide and then reducing by hydrogen in a mixture with inert gas at 493 to 505 K. The resultant catalyst contained at least 97.5% pure copper and its oxides. The surface of the catalyst particles consisted solely of copper, however. The experiments performed confirmed that the conversion of acetonitrile in the presence of a copper catalyst is not high enough. This is especially true when concentrated solutions are used. The depth of nitrile transformation in 2 hours for concentrations of 1, 5, 7, and 11.5 mol/l reached 25, 19, 6, and 3%, respectively. Certain copper salts, when added to the reaction mixture in specified amounts, were found to greatly increase the copper catalyst's activity. The best results were obtained when copper sulfate was added. At copper sulfate concentrations of 3 to  $4.5 \times 10^{-4}$  g/(mol·g), the reaction rate increased by more than a factor of 3.5 when compared with hydration in the absence of additives. Copper chloride also increased catalytic activity, albeit to a lesser extent, and copper bromide or nitrate only deactivated the catalyst. The copper catalyst studied manifested a high selectivity with respect to acetamide, i.e., a selectivity of at least 98%, both in the presence and absence of additive. Figures 4; references 5: 4 Russian, 1 Western.

### Antiadhesion Polymer Coating

917M0159C Kiev *KHIMICHESKAYA  
TEKHNOLOGIYA* in Russian No 4, Jul-Aug 91  
(manuscript received 1 Feb 91) pp 27-29

[Article by Ye.V. Sayapin, Ye.P. Frants, and V.I. Berzin, Tomsk Department, Plastopolimer Scientific Production Association, Okhtinsk]

UDC 678.743:539.612

[Abstract] Polyurethane is highly adhesive to various materials, including the metals of which molds are commonly made. For this reason, when manufacturing fluoropolymer-based polyurethane products it is necessary to create an antiadhesion interlayering between the mold and the product being molded. In view of this

need, the authors of the study reported herein developed an antiadhesion polymer coating based on the fluoroplastic F-4MBP that could be used to improve the extraction of polyurethane products from molds. Isopropyl rather than ethyl alcohol was found to be the best dispersion medium for the new coating, and the concentration of the suspension was selected by proceeding from the required thickness of a coating produced after a four- to six-layer application. The coating developed makes it possible to remove more than 1,300 products from a mold before the coating's individual sections need be repaired. Products molded in molds coated with the new coating require between 2 minutes 40 seconds and 3 minutes 15 seconds to solidify. The new coating is 20- to 25-fold more durable than its analogues, and it enables the polymer being molded within the mold to solidify faster than is possible when the new coating's conventional analogues are used. Tables 2; references 2 (Russian).

#### **Substantiating the Makeup and Main Technical Characteristics of an Experimental Coal Gas Transport Ring**

917M0159D Kiev *KHIMICHESKAYA  
TEKHNLOGIYA in Russian No 4, Jul-Aug 91*  
(manuscript received 1 Feb 91) pp 56-58

[Article by A.P. Klimenko, Yu.A. Bokserman, L.N. Ilchenko, and T.S. Dzhigirey, Gas Institute, UkSSR Academy of Sciences, Kiev]

UDC 662.69.66-663.621.547

[Abstract] In view of the need to transport great quantities of coal from the coal-recovering regions of central Siberia to the European section of the USSR, it has been suggested that a trunk pipeline be used to transport the coal required in the form of a gas flow. Because the motion of a suspension along a pipeline is very different from the motion of pure flows, the authors of the study reported herein worked to develop an experimental gas transport ring that would enable them to investigate the flow mechanism of coal suspended in a gas flow. The experimental ring developed consists of four loops with various lengths and diameters. These loops include a coal pulverization unit, a loading unit, a unit to feed and store the coal dust and carbonic acid, and a circulation unit that makes it possible to control the units' capacity and flow rate. The system developed makes it possible to use gaseous and liquid carriers, including liquid carbonic acid. The research installation makes it possible to determine the degree of compression and pressure loss required to permit the transport of coal in a suspension. Work is currently in progress to improve the system's safety by creating special safety devices to prevent the possible (albeit unlikely) formation of an explosive mixture within the pipeline.

#### **Generation of an Ultrasonic Stream of Combustion Product Plasma**

917M0159E Kiev *KHIMICHESKAYA  
TEKHNLOGIYA in Russian No 4, Jul-Aug 91*  
(manuscript received 7 Feb 91) pp 59-64

[Article by S.V. Petrov and Ye.P. Martsevov, Gas Institute, UkSSR Academy of Sciences, Kiev]

UDC 533.9:629.78

[Abstract] The use of ultrasonic streams of plasma of the combustion products of hydrocarbon gas with air affords new possibilities regarding the creation of an efficient technology for spraying protective coatings. The unique properties of ultrasonic plasma streams (their high velocities and energies, the peculiarities of their flow structure, etc.) give the plasma in the live zone properties that cannot be achieved by other methods. In view of these facts, the authors of the study reported herein performed a series of theoretical and experimental studies of the flow parameters and energy characteristics of an ultrasonic plasma generator for spraying protective coatings in a medium of combustion product plasma. It was discovered that radiation and convection are decisive for heat transfer to the plasma generator's channel walls. Consequently, the plasma generator's efficiency does not depend on the gas:air ratio; rather, it is the composition of the gas that is critical to the parameters of the plasma in the critical cross section. These facts are used as the basis for a series of computations designed to improve the plasma generator's heating of the disperse-phase particles and acceleration properties. Figures 7; references 3 (Russian).

#### **An Investigation of the Properties of Type PMS-200 High-Boiling Heat Transfer Medium**

917M0159F Kiev *KHIMICHESKAYA  
TEKHNLOGIYA in Russian No 4, Jul-Aug 91*  
(manuscript received 15 Feb 91) pp 64-67

[Article by P.I. Batura, A.F. Grebenyuk, and N.A. Dokina, Donetsk Polytechnic Institute]

UDC 621.039.534

[Abstract] The authors of the study reported herein compared the properties of different classes of substances used as high-temperature heat transfer media in the chemical, oil, and nuclear power industries. After demonstrating the superiority of methyl(ethyl)siloxane fluids over other products intended for similar purposes, the authors proceed to conduct a more in-depth examination of the polymethylsiloxane heat transfer medium PMS-200, which is produced by the Kremniypolymer [Silicon Polymer] Plant in Zaporozhye. Studies conducted on the PMS-200 indicate that it has measured viscosities of 0.200, 0.107, and 0.051 (N·s)/m<sup>2</sup> at 20, 50, and 100°C, respectively. Its density ranges from 0.97 to 0.98 g/cm<sup>3</sup>. It has a boiling point (at 1 to 3 mm Hg) of

300°C, a flash point of 300°C, and a pour point of -60°C, and its refractive index ranges from 1.390 to 1.403. It has measured heat conduction values of 0.6034, 0.5866, 0.5740, 0.5715, and 0.5531 kJ/(m·h·°C at 20, 40, 60, 80, and 100°C, respectively. Laboratory tests performed on the PMS-200 confirmed that it is a thermally stable substance and that it may be used as a heat transfer medium in heating systems. Its chemical inertness, low cost (5 to 7 rubles/kg), and sufficient raw material base make PMS-200 the preferred heat transfer medium among analogous heat transfer media used in heating installations. Tables 2; references 5 (Russian).

**The Effect of Cavitation Treatment on the Colloid Chemical Properties of Raw Material Used To Produce Commercial-Grade Carbon**

917M0159G Kiev *KHIMICHESKAYA  
TEKHNOLOGIYA* in Russian No 4, Jul-Aug 91  
(manuscript received 28 Feb 91) pp 67-71

[Article by N.N. Yaske, O.M. Yakhno, A.D. Koval, A.S. Samokhvalov, and V.V. Monyatovskiy, Kiev Polytechnic Institute]

UDC 661.666.4:665.6.002

[Abstract] Increasing the degree of use of liquid hydrocarbon raw material to produce commercial-grade carbon will increase the output of high-quality finished product and improve the ecological situation by reducing the amount of toxins released into the atmosphere. Because the quality and yield of commercial-grade carbon are known to be determined by the colloid chemical properties of the raw material, the authors of the study reported herein examined the effect that cavitation treatment has on the colloid chemical properties of the aforesaid raw material. Specifically, they studied by-product coke raw material, thermal gas oil, cracking gas oil, and mixtures of all the aforementioned. A table-model closed-loop-type cavitation unit was manufactured specifically for the studies reported herein. The following were investigated: the change in the dynamic viscosity of by-product coke raw material over time after cavitation treatment, the temperature dependence of dynamic viscosity before and after cavitation treatment, and the change in the stability factor over time given different cavitation treatment conditions. The tests conducted indicated that a cumulative cavitation effect may be used to reduce the viscosity of structured raw material composites, change their viscosity-temperature dependence, and increase the homogeneity and stratification resistance of disperse petroleum systems. Figures 4; references 6 (Russian).

### Testing Solid-Electrolyte Oxygen Sensors by Electrochemical Methods

917M0174E Moscow UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 57 No 6, Jun 91  
(manuscript received 15 May 90) pp 622-628

[Article by R.M. Guseynov, V.D. Prisyazhnyy, and B.A. Shmatko, Dagestan Pedagogical Institute, Makhachkala, General and Inorganic Chemistry Institute, UkSSR Academy of Sciences, Kiev, and Power Physics Institute, Obninsk]

UDC 541.135.4

[Abstract] Electrochemical solid-electrolyte sensors are generally used to measure the thermodynamic activity of an oxygen impurity in liquid metals. Ceramics based on zirconium dioxide poly- or monocrystals are generally used as the solid electrolyte. Problems arise when such electrolytes are used in alkaline metal coolants owing to the corrosive effect of liquid metals on the electrolyte or the formation of corrosion products on the electrolyte. In an effort to circumvent these problems, the authors of the study reported herein have proposed impedance and pulse galvanostatic methods for use in testing solid-state oxygen sensors. By calculating impedance, the authors have demonstrated the possibility of testing sensors with a liquid-metal standard electrode based on oxygen-saturated bismuth (Mo/Bi,  $\text{Bi}_2\text{O}_3//\text{ZrO}_2\cdot\text{Y}_2\text{O}_3/[\text{O}]$ , with  $[\text{O}]$  indicating the oxygen dissolved in the melt). The impedance hodograph and limiting voltage in the presence and absence of polarization of the sensor has been estimated. The value of the limiting voltage  $E(\infty)$  has been proposed as a criterion for determining the presence or absence of polarization of an electrochemical sensor in the pulse galvanostatic method presented. It has been determined that in the absence of polarization  $E(\infty)$  will equal  $IR_i$ . When a blocking layer is present,  $E(\infty)$  will equal  $I(R_i + R_p)$ . Figures 7; references 15: 11 Russian, 4 Western.

### Combined Reduction of Ti- and Ni-Type Compounds in Chloride Melts

917M0174F Moscow UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 57 No 6, Jun 91  
(manuscript received 3 Jul 90) pp 632-634

[Article by V.I. Shapoval, L.I. Zarubitskaya, Yu.V. Korobka, General and Inorganic Chemistry Institute, UkSSR Academy of Sciences, Kiev]

UDC 541.135

[Abstract] In a continuation of their research on the possibility of synthesizing intermetallic compounds of titanium with metals of the iron subgroup, the authors of the study reported herein examine the distinctive features of the electrochemical behavior of nickel and titanium when both are present in high-temperature

chloride mixtures. They have determined the potentials (stationary, anode dissolution, and cathode discharge) of titanium and nickel and used these measurements as the basis for producing powder intermetallic titanium and nickel compounds by means of electrolysis. The authors claim to be the first to make electrochemical measurements in an equimolar mixture of KCl-NaCl containing both nickel and titanium chlorides. They further state that they are the first to obtain a cathode-discharge wave for a compound of titanium and nickel. Their experiments reveal a complex pattern of the anodic dissolution of metallic nickel in melts containing both nickel and titanium compounds simultaneously. This pattern is explained by the preliminary chemical reaction of the metallic nickel with the titanium chlorides immediately after the electrode is immersed into the electrolyte. This in turn is said to result in the formation of intermetallic compounds of titanium and nickel on the nickel's surface and in the precipitation of metallic titanium. The authors thus succeed in demonstrating the possibility of high-temperature electrochemical synthesis of intermetallic titanium and nickel compounds in chloride melts. Figures 2, table 1; references 7: 5 Russian, 2 Western.

### Microstructure of a Composite Cathode for a Chemical Current Source Based on Copper Monochloride

917M0174G Moscow UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 57 No 6, Jun 91  
(manuscript received 3 Jul 90) pp 635-638

[Article by L.R. Vishnyakov, V.P. Dzeganzovskiy, V.N. Leonov, T.N. Bondarenko, G.P. Sunegin, Ye.V. Bayrachnyy, and A.B. Pospelov, Materials Science Problems Institute, UkSSR Academy of Sciences, Kiev]

UDC 621.762.4:621.3.032.2

[Abstract] The problem of replacing the silver chloride that is contained in water-activated chemical current sources by the more accessible copper chloride has become urgent. In view of the urgency of this problem, the authors of the study reported herein investigated the microstructure of a cathode material based on cast copper chloride. The material studied includes copper chloride as its active mass and two functional additives. The first of these,  $\text{SnCl}_2$  in the amount of 1% by weight, is used as a reducing agent. The second is an electricity-conducting additive ( $\text{Me}_n\text{S}$ ). Together with the reinforcing copper mesh, the additives form a continuous skeleton in the material. This skeleton provides electronic conduction and better conditions for electrical discharge of the material in a chemical current source. Through their research, the authors succeeded in determining that the composition and concentration of electricity-conducting additives and the speed of the crystallization process are key to giving a cast copper chloride electrode the optimal microstructure. Figures 3; references 7: 6 Russian, 1 Western.

### Effect of pH and Length of Storage of Water Samples on Atom-Absorption Determination of Mercury

917M0156A Kiev *KHIMIYA I TEKHOLOGIYA VODY* in Russian Vol 13, No 3, Mar 1991 pp 227-230

[Article by V. P. Antonovich, I. V. Bezlutskaya, Yu. V. Zelyukov and M. M. Novoselova; Physico-chemical Institute imeni A. V. Bogatskiy; UkSSR Academy of Sciences; Odessa]

UDC 543.3.546.49

[Abstract] A study of the effect of the pH and length of storage of water samples on the completeness of reduction of mercury (II) by tin (II) chloride, an explanation of the nature of this effect and proposal of means of eliminating it involved determination of mercury (II) introduced into samples of deionized, bidistilled and tap waters. Tin (II) chloride, widely used in atom-absorption determination of inorganic forms of mercury in natural waters, did not reduce mercury from its hydrocomplexes. Comparison of redox potentials of  $E^0\text{Hg}(\text{OH})_2/\text{Hg}=0.26$  V and  $E^0\text{Sn}_{(\text{IV})}/\text{Sn}_{(\text{II})}=0.15-0.21$  V pairs ( $E^0\text{Hg}_{(\text{II})}/\text{Hg}=0.85$  V) confirmed this. Redox reaction was complete at a difference of reacting redox-pair potentials of at least 20 V. Use of nitric acid (0.17 M) to preserve natural water samples stabilized tin(II)-chloride reduced mercury within 7 days but did not break down mercury hydrocomplexes previously existing in waters. The possibility of using tin(II) chloride for atom-absorption determination of inorganic mercury forms in natural waters after preliminary treatment of water samples with hydrochloric acid ( $C_{\text{HCl}}$  greater than but almost equal to 2.7 M) for 2-3 hours was demonstrated. Figure 1; references 12: 8 Russian; 4 Western.

### Chemiluminescent Method of Determining Nitrates and Nitrites in Waters With Use of Gas Extraction of Nitrogen Dioxide

917M0156B Kiev *KHIMIYA I TEKHOLOGIYA VODY* in Russian Vol 13, No 3, Mar 1991 pp 230-234

[Article by A. T. Pilipenko and O. V. Zuy; Institute of Colloid Chemistry and Chemistry of Water imeni A. V. Dumanskiy; UkSSR Academy of Sciences; Kiev]

UDC 543.3:535.379

[Abstract] A chemiluminescent method was developed to determine nitrites and nitrates in waters by using gas extraction of  $\text{NO}_2$  in a luminol solution. Determination of nitrites involved their decomposition by sulfuric acid, extraction of the nitrogen oxides formed from the aqueous solution by a purified air flow and, then, detection in the carrier gas flow by the  $\text{NO}_2$  reaction with luminol. The limit of detection of  $\text{NO}_2$  in natural waters was 0.03 mg/l. Determination of the overall  $\text{NO}_2$ - and  $\text{NO}_3$ - involved preliminary passage of the water being analyzed through a regulator containing copper-plated

cadmium to reduce the  $\text{NO}_3$ - to  $\text{NO}_2$ - and determination of the sum of  $\text{NO}_2$ -+ $\text{NO}_3$ - by the reaction of  $\text{NO}_2$ - with luminol.  $\text{NO}_3$ - was determined by the difference. Figures 3; references 16: 9 Russian; 7 Western.

### Treatment of Livestock Sewage by Membrane Methods

917M0156C Kiev *KIMIYA I TEKHOLOGIYA VODY* in Russian Vol 13, No 3, Mar 1991 pp 252-255

[Article by V. V. Kotov, V. O. Chernyshov, Ye. V. Pronina et al.; Voronezh Agricultural Institute]

UDC (66.087.97+66.067.38):541.135

[Abstract] A study of mechanisms of combined ultrafiltration-electrodialysis treatment of the liquid fraction of livestock sewage showed that ultrafiltration may provide more than 80 percent removal of total phosphorus from the runoff and 90 percent removal of protein and total removal of microorganisms from the dry substance. Technological methods of regeneration of ultrafiltration membranes were discussed. Treatment of the filtrate by electrodialysis with ion-exchange membranes reduced its salt content by 75-90 percent. After such treatment, the remaining water may be used for irrigation purposes or for washing livestock premises. Figures 2; references 6: 5 Russian; 1 Western.

### Ultrafiltration Concentration of Scandium From Multicomponent Solutions

917M0156D Kiev *KHIMIYA I TEKHOLOGIYA VODY* in Russian Vol 13, No 3, Mar 1991 pp 255-259

[Article by K. A. Volchek, Yu. I. Dytnerkiy, A. Al-Naif et al.; Moscow Chemico-Technological Institute imeni D. I. Mendeleev; Institute of Geochemistry and Analytical Chemistry imeni V. I. Vernadskiy; USSR Academy of Sciences; Moscow]

UDC 661.879.063.94:66.067.38

[Abstract] The article confirmed the possibility of selective extraction of scandium from multicomponent solutions by ultrafiltration in combination with chelation. The method involves selective binding of ions of metals in complexes or aggregates with water-soluble polymers and subsequent ultrafiltration, in the course of which unbound components of the solution are separated from the complexes or aggregates. Conditions of scandium binding were determined and regeneration of the polymer complexing agent was studied. A procedure of ultrafiltration concentration of scandium from underground mineralized waters was proposed. Figures 5; references 9: 7 Russian; 2 Western.

**Use of Biologically Treated Sewage in Recirculated and Drainless Water Supply of Industrial Enterprises in Ukraine**

917M0156E Kiev KHIMIYA I TEKHOLOGIYA  
VODY in Russian Vol 13 No 3, Mar 1991 pp 260-264

[Article by A. M. Koganovskiy, L. N. Gora, V. Ya. Romantsova and T. M. Tkachuk; Institute of Colloid Chemistry and Chemistry of Water imeni A. V. Dumanskiy; UkSSR Academy of Sciences; Kiev]

UDC 628.33+628.543

[Abstract] Treatment of biologically treated sewage by adsorption filtration through active anthracite and Na-cation exchange softening produced water with COD of 10-12 mgO/l, permanganate oxidizability of less than but almost equal to 3.5-4 mgO/l, chromaticity )Cost of such water produced from biologically treated municipal sewage did not exceed the cost of production of process water of equal quality from natural fresh water supply sources. Use of adsorption purified and softened municipal sewage for replenishing circulating heat exchange water supply systems ensured the limitation of stabilization discharge and other irreversible losses (in addition to losses from evaporation and droplet wind carry-over), not exceeding 2 percent. This guaranteed that the quantity of biologically treated sewage discharged into Ukrainian reservoirs at present will meet the needs for makeup water for recirculating water systems of all Ukrainian industrial enterprises. References 9: 8 Russian; 1 Western.

**Technology of Mg-Na-Cation-Exchange Softening of Natural Waters and Sewage With Recuperation of Spent Solutions**

917M0156F Kiev KHIMIYA I TEKHOLOGIYA  
VODY in Russian Vol 13, No 3, Mar 1991 pp 264-269

[Article by L. N. Poletayev, M. M. Agamaliyev, E. G. Mamedova and Sh. M. Manafov; Azerbaijan Institute of Petroleum and Chemistry imeni M. Azizbekov; Baku]

UDC 628.387.621.187

[Abstract] A study of the process of Mg-Na-cation-exchange softening of slightly salty waters during regeneration of ion-exchange filters by sulfite solutions in a closed circuit was described and discussed. Regression equations for practical calculations of the process were obtained. A fundamental flow diagram was proposed to increase the capacity of the cation exchangers by Ca<sup>2+</sup>, exclude lime expenditure on Mg<sup>2+</sup> sedimentation and simplify and cheapen the process of recuperation of CaSO<sub>4</sub> sediment. Figures 6; references 5 (Russian).

**Biological Activity of Waters Containing Surfactants**

917M0156G Kiev KHIMIYA I TEKHOLOGIYA  
VODY in Russian Vol 13, No 3, Mar 1991 pp 270-283

[Article by S. A. Ostroumov; State Institute imeni M. V. Lomonosov; Moscow]

UDC 574.64:556.531.4

[Abstract] A survey of some recent studies of the effect of surfactants and aquatic environments polluted by surfactants on the organism served to analyze the ecological danger of pollution of the aquatic environment by different surfactants and involved assessment of the effect of an aquatic environment containing surfactants on different test organisms. The survey included some of Ostroumov's experimental data. The survey showed the significant danger of surfactants and surfactant-containing substances as pollutants of the hydrosphere and biosphere. Surfactants and surfactant-containing substances include a very heterogeneous group of compounds in the level of their biological activity and in the diversity of their effect on living organisms. The study recommended expansion of the spectrum of test-organisms and test-functions used in biological diagnosis and biological testing of surfactants and aquatic environments. The study confirmed the effectiveness of use of non-traditional test-objects and new methods in determining hazards from surfactants. Efforts to reduce the amount of surfactants in sewage and polluted waters were recommended. The author's experimental data complemented information found during the survey and confirmed it. References 83: 40 Russian; 43 Western.

**Adsorption Purification of Chemical Enterprise Sewage With Use of Combination of Different Types of Adsorbents**

917M0166A Kiev KHIMIYA I TEKHOLOGIYA  
VODY in Russian Vol 13, No 6, June 1991 pp 503-506

[Article by V. V. Podlesnyuk, L. Ye. Fridman, T. Ye. Mitchenko et al.; Institute of Colloid Chemistry and Chemistry of Water imeni A. V. Dumanskiy; UkSSR Academy of Sciences; Kiev]

UDC 628.31

[Abstract] A study of the use of a combination of different types of sorbents in the flow diagram of treatment of a chemical enterprise sewage containing a mixture of benzene derivatives, organic acids, pesticides and phenols used combinations of active anthracite, porolas T and ion-exchange resin VP-1-AP. Use of different combinations of the adsorbents revealed the optimal combination for use in sewage treatment. A combination of active anthracite - porolas T - ion-exchange resin VP-1-AP - active anthracite proved to be most effective in sewage treatment with use of the eluent method of

regeneration of porolas T and ion-exchange resin by acetone. Figure 1; references 16: 11 Russian; 5 Western.

**Analysis of Current Flow Diagrams of Tertiary Treatment of Biologically Purified Municipal and Industrial Sewage For Purpose of Use For Needs of Industrial Water Supply**

917M0166B Kiev *KHIMIYA I TEKHOLOGIYA VODY* in Russian Vol 13, No 6, June 1991 pp 511-516

[Article by A. M. Koganovskiy and T. M. Tkachuk; Institute of Colloid Chemistry and Chemistry of Water imeni A. V. Dumanskiy; UkSSR; Kiev]

UDC 628.33+628.543

[Abstract] A survey of the literature concerning flow diagrams of tertiary treatment of biologically purified sewage included discussion of 4 methods of treatment: 1) use of coagulants, lime and flocculants for tertiary treatment of biologically purified municipal waters; 2) use of ozone and other oxidants for tertiary treatment of biologically purified sewage; 3) use of biosorption for improving the quality of municipal sewage and 4) use of ultrafiltration for tertiary treatment of biologically purified sewage. The survey showed that most countries use, for tertiary treatment of sewage, a complex multi-stage technology including both reagent treatment (lime pretreatment, coagulation, flocculation and ozonization) and the use of physico-chemical processes (ultrafiltration, ion-exchange, adsorption and reverse osmosis). Treatment of sewage before discharge into a reservoir is deeper than treatment of water for reuse and usually includes reverse osmosis demineralization. In spite of the relatively high cost and complexity of tertiary treatment of sewage (25-36 cents/m<sup>3</sup>) the water produced is used only for industrial and agricultural purposes. Studies of the technical and economic advisability of producing, from sewage, waters for more complex technological needs were found to be scarce and to involve, for the most part, pilot studies. Large-size plants are, basically, still in the design stage. References 43: 14 Russian; 29 Western.

**Problem of Residual Aluminum in Treated Water**

917M0166C Kiev *KHIMIYA I TEKHOLOGIYA VODY* in Russian Vol 13, No 6, June 1991 pp 517-534

[Article by I. M. Solomentseva, L. A. Velichanskaya and I. G. Gerasimenko; Institute of Colloid Chemistry and Chemistry of Water imeni A. V. Dumanskiy; UkSSR Academy of Sciences; Kiev]

UDC 628.16+628.344

[Abstract] A survey of the literature provided an examination of medical, biological and technological aspects of high concentrations of residual aluminum in treated tap water. It discussed norms of aluminum content in drinking water in the Soviet Union and abroad. The

discussion emphasized examination of the dangers of high levels of aluminum in treated water, transformations of forms of aluminum in solution and in treated water, the effect of different factors on the amount of residual aluminum and methods of reducing the residual aluminum content. Figures 16; references 114: 45 Russian; 69 Western.

**Substantiation and Basic Technical and Economic Indicators of New Drainless Method of Demineralizing Water by Ion Exchangers**

917M0166D Kiev *KHIMIYA I TEKHOLOGIYA VODY* in Russian Vol 13, No 6, June 1991 pp 538-544

[Article by A. V. Mamchenko and T. I. Yakimova; Institute of Colloid Chemistry and Chemistry of Water imeni A. V. Dumanskiy; UkSSR Academy of Sciences; Kiev]

UDC 628.543

[Abstract] Substantiation of the principle of creating a new drainless method of demineralizing water by ion exchangers was described and discussed. The method combined the advantages of the reagent method of water demineralization by means of H<sup>+</sup>-OH<sup>-</sup> or H<sup>+</sup>-HCO<sub>3</sub><sup>-</sup> ionization with a high degree of demineralization and reagent-free methods (Siroterm and Kariks) which practically exclude expenditures of reagents and permit procurement only of salts found in the initial water in the form of water treatment wastes. Basic technical and economic indicators of the new method of water demineralization were assessed and compared with characteristics of drainless variants of known technological processes which produce water with the same deep demineralization. References 20: 11 Russian; 9 Western.

**Immobilization of Destroyer Bacteria on Artificial Fibers For Removing Anionic Surfactants From Water**

917M0166E Kiev *KHIMIYA I TEKHOLOGIYA VODY* in Russian Vol 13, No 6, June 1991 pp 548-554

[Article by S. S. Stavskaya, I. I. Shamolina, G. N. Nikovskaya et al.; Institute of Colloid Chemistry and Chemistry of Water imeni A. V. Dumanskiy; UkSSR Academy of Sciences; Kiev; Leningrad Institute of Textile and Light Industry imeni S. M. Kirov]

UDC 576.6.695

[Abstract] A study of the interaction of anionic surfactants of alkylsulfates, alkylsulfonates (wolginate), alkylbenzolsulfonates with synthetic fibers used bacteria strains *Pseudomonas aeruginosa* 1 C, *P. rathonis* T and *P. alcaligenes* TR. Isotherms of sorption on fibers were constructed. Kinetics of sorption and desorption of bacteria in solutions of anionic surfactants were studied. Activation of the fibers by chemical reagents increased their affinity for the bacteria cells but the degree of

affinity of bacteria for the fibers differed in different strains of destroyer bacteria of anionic surfactants. The immobilized bacteria preserved their capacity to destroy anionic surfactants and stimulation of the destructive activity in the presence of the fibers occurred in some cases. The absence of harmful effects of the fibers on the destructive activity of the bacteria permits their use as carriers in processes of microbiological removal of anionic surfactants from water. Figures 4; references 20: 19 Russian; 1 Western.

**Effect of Chemical Factors in Water on Preservation of Adhesiveness of Enteropathogenic Bacteria**

917M0166F Kiev *KHIMIYA I TEKHOLOGIYA VODY* in Russian Vol 13, No 6, June 1991 pp 570-574

[Article by L. V. Grigoryeva and G. I. Korchak; Republican Scientific Hygiene Center; Ministry of Health UkSSR; Kiev]

UDC 628.322:614.3

[Abstract] A study of the effect of some chemical factors in water on preservation of adhesive activity of pathogenic Eschericia and Salmonella strains used 24 strains (10 strains of Salmonella and 14 strains of pathogenic Eschericia) isolated from objects in the environment, especially sewage and reservoirs, and one museum strain as a control. The study included consideration of a high concentration of salts, pH and active chlorine. Salmonella adhesions were more resistant to the effect of 0.1-0.3 mg/l of chlorine in tap water. Adhesions of pathogenic Eschericia displayed the greatest resistance to the effect of pH 7.0 and 8.8 of tap water and sea water. The findings proved to be significant in assessment of the epidemiological risk of the water factor in transmission of intestinal infections during conditioning of potable water. Figures 2; references 15: 5 Russian; 10 Western.

### Distinctive Features of the Dynamics of Damage to Semiconductor Surfaces by Nanosecond Laser Pulses

917M0171A Moscow *POVERKHNOST: FIZIKA, KHIMIYA, MEKHANIKA* in Russian No 6, Jun 91 (manuscript received 30 Oct 89; after revision 22 May 90) pp 86-92

[Article by G.M. Gusakov, A.A. Komarnitskiy, and A.S. Em, Moscow Institute of Electronic Technology]

UDC 621.373.826:536.421.1

[Abstract] Time-resolved reflectivity measurements and optical microscopy were used to examine the dynamics of damage inflicted on Si, Ge, and GaAs surfaces by laser pulses. Specifically, the authors used specimens of monocrystalline Si and Ge with an orientation of (111) and specimens of GaAs with an orientation of (100). Two pulse modes were used: 1)  $\lambda_1 = 0.53 \mu\text{m}$ ,  $\tau_{p1} = 50 \text{ ns}$ ; and 2)  $\lambda_2 = 1.06 \mu\text{m}$ ,  $\tau_{p2} = 50 \text{ ns}$ . The first initiated the formation of a liquid phase in the thin surface layer of the semiconductor, and the second acted upon the already-fused, highly absorbent layer. The heating rates of a semiconductor melt subjected to nanosecond laser irradiation obtained from the authors' experiments turned out to be much less than those predicted by the thermal model based on a nonlinear Stefan problem. The effect of the degree of overlapping of the laser spots on the dynamics of the process of nanosecond pulsed laser irradiation that the authors discovered could not be explained by the simple thermal model of the heating and melting of semiconductors. Possible ways of improving the model to more adequately describe the dynamics of the transition to surface damage as the laser pulses' intensity increases are discussed, but no definitive conclusion is drawn. Figures 3; references 18: 6 Russian, 12 Western.

### Reproducibility of the Composition and Properties of Films of Tungsten-Titanium Alloy Produced by Magnetron Sputtering of Composite Targets

917M0171B Moscow *POVERKHNOST: FIZIKA, KHIMIYA, MEKHANIKA* in Russian No 6, Jun 91 (manuscript received 22 Feb 90; after revision 9 Oct 90) pp 107-112

[Article by V.Yu. Yashchak, V.G. Glebovskiy, A.I. Dudarchik, A.P. Dostanko, V.V. Baranov, and V.V. Kolos, Minsk Electronics Institute]

UDC 539.23:P669.27.849

[Abstract] In view of the promise of W-Ti alloy as a material for use as the barrier layer of VLSI circuits, the authors of the study reported herein studied the reproducibility of the composition and properties of films of the alloy W-Ti obtained by magnetron sputtering of composite targets onto Si plates with test structures of Schottky TTL VLSI circuits. The method of Auger

electron spectroscopy was used to investigate the distribution of elements throughout the depth of W-Ti layers with a thickness of  $0.18 \pm 0.02 \mu\text{m}$  and a specific resistance not exceeding 5  $\Omega$  per square. The films' composition was monitored by spectral analysis, and the reproducibility of the films' composition throughout its depth was checked by electron Auger profiling on an OZIOS-10-005 unit. Spectral analysis revealed that the composition of films sputtered onto a composite target (particularly in the initial period of their use and after 130 sputtering cycles) contains about 19% (by weight) Ti. Tungsten was found to account for the remainder of the films' composition, and the deviation from this ratio during the course of the films' use never exceeded 2%. The quantitative relationship of Ti and W was found to remain virtually constant throughout the films' entire thickness (with the exception of the surface layer): about 42 atomic percent Ti and 50 atomic percent W. At the moment of the jump, however, the W concentration was observed to increase to about 55 atomic percent. A transition layer of some thickness was seen to form at the W-Ti/SiO<sub>2</sub> interface. Interpretation of this phenomenon was complicated by the large error resulting from the small boost charge of the specimens when the Auger spectra were recorded. There was, however, an obvious trend toward a change in concentration of the components Si, Ti, and O throughout the depth of the layer. The properties of elements with the structure of W-Ti/Al layers, specifically junctions with a Schottky barrier to the n-Si layer with a PtSi substrate about 40 nm thick, were found to depend not only on the reproducibility of the catalyst of the W-Ti alloy film, but also on its application rate, which is in turn dictated by the power fed to the magnetron source. Si/PtSiW-Ti/Al Schottky diodes are characterized by higher current leakage levels than are Si/PtSi/Mo/Al diodes. In neither case, however, do the current leakages prevent the diodes' use at Schottky TTL integrated circuits. The phenomenon of silicide formation in the case where the PtSi/W-Ti interface is held at 450° is observed to occur slowly and is related to the presence of the structure of the solid solution of the alloy W-Ti and the concentration of interstitial impurities such as carbon in the transition layer. Figures 3; references 7: 5 Russian, 2 Western.

### Alloying the Surface Layer of Iron by the Method of Ion Mixing

917M0171C Moscow *POVERKHNOST: FIZIKA, KHIMIYA, MEKHANIKA* in Russian No 6, Jun 91 (manuscript received 18 Sep 89; after revision 2 Feb 90) pp 113-120

[Article by L.P. Chupyatova, I.A. Kmarovskiy, T.Ye. Peksheva, and I.G. Murzin, Moscow Steel and Alloys Institute]

UDC 669.018.6

[Abstract] The authors of the study reported herein used the methods of Rutherford backscattering and Auger

electron spectroscopy to study the surface doping of iron with Nb, W, Ni, Al, and Cu by the technique of ion mixing. Specimens of iron with a grain size of 300  $\mu\text{m}$  were polished until a final grain dispersion of 0.6  $\mu\text{m}$  was achieved. The specimens were then subjected to chemical neutralization and ion etching at a residual pressure of  $10^{-4}$  Pa. The specimens were coated by the magnetron sputtering method on an Alcatel SCM-650. The authors experimentally determined the values of mixing effectiveness with  $\text{Ar}^+$  ions at a current density of about 0.8  $\mu\text{A}/\text{cm}^2$  and  $\text{Xe}^+$  ions at a current density of 0.2  $\mu\text{A}/\text{cm}^2$  in the following two-layer systems at ambient temperature: Fe-W, Fe-Nb, Fe-Ni, Fe-Al, and Fe-Cu. In the systems Fe-Nb and Fe-W, mixing effectiveness was found to be a function of ionic current density when  $J > 0.2 \mu\text{A}/\text{cm}^2$ . It was further concluded that in the system Fe-Ni mixing occurs by the route of development of linear cascades. In the systems Fe-Nb and Fe-W, mixing was determined to occur by the route of the development of thermal peaks, and in the systems Fe-Al and Fe-Cu it was radiation-stimulated diffusion that played the decisive role. In the case of high radiation defect mobility, it was the magnitude and sign of  $\Delta H$  (the mixing heat) that dictated the process of the elements' propagation (either facilitating it as in the system Fe-Al or impeding it as in the system Fe-Cu). In the case of low defect mobility, however, mixing within the systems studied was found to depend not on  $\Delta H$  but rather on the ratio of the masses of the system's bombarding ions and atoms, which in turn dictates cascade density. Figures 5, tables 2; references 13: 2 Russian, 11 Western.

#### X-Ray Photoelectron Spectrum Study of the Ratio and State of Atoms on the Surface of the Superconducting Composite Y-Ba-Cu-Ru-O

917M0171D Moscow *POVERKHNOST: FIZIKA, KHIMIYA, MEKHANIKA* in Russian No 6, Jun 91 (manuscript received 12 Sep 89; after revision 11 Apr 90) pp 121-126

[Article by Yu.M. Shulga, V.I. Rubtsov, E.N. Izakovich, Yu.G. Borodko, Chemical Physics Institute USSR Academy of Sciences, Chernogolovka]

UDC 542

[Abstract] Ruthenium is promising as a modifier of  $\text{YBa}_2\text{Cu}_{7-8}$  superconductors because it can be added to the starting charge in rather large quantities without reducing its critical temperature ( $T_c$ ). For this reason, the authors of the study reported herein examined the state and ratios of elements on the surface of the superconducting composite  $\text{YBa}_2\text{Cu}_{2-7}\text{Ru}_{0.3}\text{O}_x$ . The study specimens were obtained by sintering a mixture of  $\text{Y}_2\text{O}_3$ ,  $\text{BaCO}_3$ ,  $\text{CuO}$ , and  $\text{RuO}_2$  (or Ru) in air at  $880^\circ$  for 16 hours. The resultant powder was pressed into tablets measuring  $20 \times 8 \times 0.5 \text{ mm}^3$  and annealed in an oxygen medium for 6 hours at  $880^\circ$ . Also produced were non-superconducting composites with the composition  $\text{YBa}_2\text{Cu}_{2.2}\text{Ru}_{0.8}\text{O}_y$  and  $\text{RuO}_2$ . It was discovered that

both specimens of the system Y-BaOCu-Ru-O had surfaces that were depleted of copper and enriched with barium and ruthenium. The ruthenium in both compositions was found to be characterized by a high degree of oxidation (most likely  $\text{Ru}^{8+}$ ). Unlike the compound containing  $\text{RuO}_4$ , the one containing  $\text{Ru}^{8+}$  was observed to form a separate phase that crystallizes in the form of smaller particles when compared with the main superconducting phase  $\text{YBa}_2\text{Cu}_{7-8}$ . Figures 3, table 1; references 24: 6 Russian, 18 Western.

#### A Study of the Effect of a Beam of Medium-Energy Nitrogen Ions on the Surface of Composite Materials

917M0171E Moscow *POVERKHNOST: FIZIKA, KHIMIYA, MEKHANIKA* in Russian No 6, Jun 91 (manuscript received 18 Apr 90; after revision 16 Jun 90) pp 127-131

[Article by R.B. Luban, P.A. Verkhovodov, R.I. Kryzhanovskaya, M.A. Negodayev, and A.V. Bagulya, Institute of Materials Science Problems, UkSSR Academy of Sciences, Kiev, and Physics Institute, USSR Academy of Sciences, Moscow]

UDC 537.534.9;669.018.95

[Abstract] The authors of the study reported herein examined the effect of an 18-keV nitrogen ion beam on the surface of copper-based composites containing  $\text{LaB}_6$  impurities. All of the specimens studied were produced by powder metallurgy techniques and had porosities ranging from 1 to 5%. The specimens were irradiated on a GELIS ion accelerator. Throughout the entire series of study measurements, the specimens were kept at a temperature not exceeding  $100^\circ$ . It was established that  $\text{LaB}_6$  inclusions cause a decrease in composites' sputtering coefficient and an increase in their ion electron emission coefficient.  $\text{LaB}_6$  inclusions were also found to stimulate the growth of "cones" on the cathode surface (this was attributed to the more intensive sputtering of copper). The authors derive and present expressions for estimating the values of each of these coefficients. The expressions have been found to yield results that are in satisfactory agreement with experimental data. Figures 5; references 6 (Russian).

#### Diagnosing Superconductivity of High-Temperature Superconductors Based on Their Surface Luminescence Spectra

917M0171F Moscow *POVERKHNOST: FIZIKA, KHIMIYA, MEKHANIKA* in Russian No 6, Jun 91 (manuscript received 14 Aug 89; after revision 25 Jan 91) pp 132-139

[Article by I.Ya. Fugol, V.N. Samovarov, and Yu.I. Rybalko, Low Temperatures Physical Technical Institute, UkSSR Academy of Sciences, Kharkov]

UDC 538.945

[Abstract] The authors of the study reported herein conducted comprehensive cathode luminescence, resistive, and magnetic studies of high-temperature superconductors of the general formulas  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  and  $\text{La}_{2-x}\text{Ba}_x\text{CuO}_{4-\delta}$ . They also compared the luminescence spectra of the aforesaid high-temperature superconductors, metals, and dielectrics. The studies confirmed that the multicomponent luminescence spectra of high-temperature superconductors is largely dictated by their surface illumination centers, which are absorption centers by nature. Those components of the spectrum (the  $\alpha$ -line and continuum at 450 nm) that sense the system of free charge carriers were established to exert an effect on the superconducting phase. They were found to be characteristic of the spectra of metals but absent in those of dielectrics. Negative atomic and molecular oxygen ions and neutral oxygen molecules were deemed the most likely surface centers. It was also discovered that oxygen can move from a specimen's bulk to its surface as a result of excitation (this avenue is especially open in the case of high-temperature superconductors) or else as a result of chemisorption from the environment. The nature of the interaction between a specimen's bulk and surface and the environment was found to undergo critical changes at the superconductivity transition, at which time critical changes in the charge and lattice subsystems of high-temperature superconductors occur. The authors attributed the sensitivity of surface centers to the superconducting phase to this fact. Figures 6; references 17: 13 Russian, 4 Western.

### Two-Component Attenuation of Landau Quantization of 2D-Electrons in $\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$ Heterostructures

917M0171G Moscow *POVERKHNOST: FIZIKA, KHIMIYA, MEKHANIKA* in Russian No 6, Jun 91 (manuscript received 23 Apr 90; after revision 31 Aug 90) pp 156-158

[Article by V.I. Kadushkin, V.A. Kulbachinskiy, and A.P. Senichkin, Physics Department, Moscow State University imeni M.V. Lomonosov]

UDC 621.317:539.211

[Abstract] The nonthermal, collision expansion of Landau levels is one of the factors limiting the amplitude of Shubnikov-de Haas oscillations. In a continuation of research on this topic, the authors of this concise report have studied the two-component attenuation of Landau quantization of 2D-electrons in  $\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$  heterostructures. Specifically, they measured the oscillations of transverse magnetic resistance at different temperatures and different temperature dependences of conduction. The test specimens were grown by the method of molecular beam epitaxy, and the conventional experimentation methodology was used. The experiments conducted led the authors to conclude that the experimental dependences  $T_D(T)$  ( $T_D$  being the

Dingle temperature) obtained from measurements of the attenuation of the Shubnikov-de Haas oscillations do not agree with quantitative estimates of the fit parameters for either the two- or three-dimensional cases. This led the authors to conclude that the expansion mechanism of Landau levels is much more complex for 2D-electrons than for the three-dimensional case. The distinctive features of the aforementioned heterostructures with 2D-electrons are discussed briefly. Figures 2; references 10: 4 Russian, 6 Western.

### Picosecond Photoconduction of Thin CdSe Films

917M0171H Moscow *POVERKHNOST: FIZIKA, KHIMIYA, MEKHANIKA* in Russian No 6, Jun 91 (manuscript received 15 Feb 90; after revision 31 Aug 90) pp 158-160

[Article by R. Tomashyunas, M. Pyatrauskas, Yu. Vaytkus, A. Zhindulis, I. Kutra, and R. Masteyka, Vilnius University]

UDC 537.311.3

[Abstract] Cadmium chalcogenides enjoy a good deal of popularity as photodetectors of visible light. In polycrystalline layers, their charge carriers may have a life on the order of nanoseconds. Achieving such and even shorter photoconduction relaxation times requires eliminating the effect of charge carrier adhesion. In an effort to solve this problem, the authors of this concise report conducted an in-depth analysis of the picosecond photoconduction of polycrystalline CdSe films. The studied CdSe films that were sputtered onto a pyroceram substrate with platinum electrodes that was heated up to 530 K. Their studies of charge carrier mobility proved to offer new insight into conduction in polycrystalline films. Analyses of such conduction generally assume that charge carrier mobility decreases relative to the monocrystal by about  $\exp(-\phi_s/kT)$ , i.e., primarily owing to the effect of the potential intercrystallite barrier. At high excitation levels, however, the potential intercrystallite barriers are completely "extinguished." The charge carrier mobility determined in the studies reported here amounted to  $\mu \approx 8 \text{ cm}^2/(\text{V}\cdot\text{s})$ , which is two orders of magnitude less than in a monocrystal ( $\mu_k \approx 800 \text{ cm}^2/(\text{V}\cdot\text{s})$ ). This fact led the authors to conclude that mobility is determined by the scattering of the charge carriers on the intercrystallite interlayerings. Figure 1; references 5 (Western).

### Purifying Metallurgical Silicon

917M0172A Moscow *IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY* in Russian Vol 27 No 4, Apr 91 (manuscript received 28 Jun 89) pp 663-665

[Article by V.N. Lozovskiy, N.F. Politova, O.V. Lastushkina, and B.P. Masenko, Novochoerkassk Polytechnic Institute imeni Sergo Ordzhonikidze]

UDC 546.28.05

[Abstract] In the past few years metallurgical silicon has enjoyed use as a construction material in the production of a wide array of products and instruments. Attempts to use metallurgical silicon as an inexpensive material for special-purpose instruments have dramatically increased demands for the production and purification of commercial grades of silicon. The existing purification methods are all plagued by the common shortcomings of low productivity and high cost. In view of these shortcomings, the authors of the study reported herein have worked to develop a comparatively inexpensive and productive purification technique intended for use in producing silicon that is of a lower purity class than semiconductor-grade silicon but that is nevertheless suitable for use in producing both various general-purpose instruments and special-purpose instruments (after additional purification). In essence, the new three-stage procedure entails dissolving the silicon in aluminum, transferring the impurities to the melt, cooling the melt, precipitating out the silicon crystals, and washing the aluminum off the silicon in acids. Purification of the silicon occurs primarily by crystallization displacement of the impurities into the melt as the crystals grow (stage 2). Further purification results from dissolution of the impurities in the aluminum (stage 1) and the subsequent acid washing (stage 3). The authors establish that the effectiveness of purification depends on the composition of the Al + Si alloy, the grain size of the purified power, and the composition of the solvents. The new technique makes it possible to raise the purity level of silicon from 98.0-99.0% (by weight) to 99.9% (by weight). Subsequent crystallization purification by growing an ingot from the melt can raise the product's purity level to 99.99% Si (by weight). References 4: 2 Russian, 2 Western.

#### Producing Amorphous Hydrogenated Si Films in Multielectrode System

917M0172B Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 14 Jun 89) pp 666-671

[Article by M.M. Mezdrogina, O.A. Golikova, M.M. Kazanin, V.Kh Kudoyarova, G.B. Yushka, K.L. Arlauskas, A.F. Bardamid, R.A. Khaydarov, and K.L. Sorokina, Physical Technical Institute imeni A.F. Ioffe, USSR Academy of Sciences]

UDC 546.28'11

[Abstract] The method of high-frequency breakdown of silane-containing gas mixtures is one of the main ways of obtaining films of hydrogenated silicon ( $\alpha$ -Si:H). In view of the promise of  $\alpha$ -Si:H as a material for use in creating a wide range of electronic instruments, the authors of the study reported herein examined the effect that changing the design of the plasma interval used in producing  $\alpha$ -Si:H films has on the plasma chemical reaction and, consequently, on the physicochemical properties of the

films produced. Specifically, they considered the changes on the following properties: the optical width of the forbidden band, photo- and dark conduction, electric conduction activation energy, specific shift of the electrons and holes, density of the localized states in the mobility gap, and composition of the  $\alpha$ -Si:H films (i.e., their hydrogen content and type of hydrogen-silicon bond). Experiments were conducted to determine the effect of producing amorphous hydrogenated silicon in diode and multielectrode systems. Changing the design of the plasma interval did indeed change the type of homogeneous reactions occurring in the interval. This change resulted in an increase in the relative concentration of long-lived radicals and mobility of the adatoms on the substrate surface. This increase in adatom mobility in turn resulted in a decrease in microstructural inhomogeneity and in a decrease in the density of the localized states in the mobility gap. Process parameters (including substrate temperature and film application rate) were found to have a less pronounced effect when a multielectrode system was used to produce  $\alpha$ -Si:H films than when a diode system was used. Figures 5; references 3: 2 Russian, 1 Western.

#### The Effect of Thin Organosilicon Films on the Physicochemical State of an Oxidized Silicon Surface

917M0172C Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 18 May 89) pp 672-674

[Article by O.A. Serdyuk, E.A. Kabanova, V.N. Matveyenko, T.D. Lisovskaya, and S.A. Gurov, Moscow State University imeni M.V. Lomonosov]

UDC 547.25+537.311.3

[Abstract] The authors of the study reported herein examined the effect of treating an oxidized silicon surface with thin organosilicon films. The axisymmetric bending method was used to study the mechanical strength of oxidized silicon wafers in the presence and absence of adsorption films of different organosilicon films on their surfaces. Wafers of type KEF-0.05 monocrystalline silicon with an orientation in the plane (111) and an initial growth dislocation density of  $10^{-4}$  cm<sup>-2</sup> were used for the study. The wafers were 250 +/- 10  $\mu$ m thick. An oxide film with a thickness of 0.5 +/- 0.05  $\mu$ m was formed on the surface of each specimen by the combined oxidation method (in dry air and moist and dry oxygen at 1,100°C). The breaking loads discovered during the experiments on specimens with a thermal oxide film were clustered on two levels. The first (bottom) level was found to correspond to the brittle fracture loads for crystalline silicon, and the second (top) level exceeded these loads by a factor of 3 to 3.5. Specimens subjected to treatment with organosilicon compounds did not manifest this two-level breaking load pattern. In essence, no lower level was discovered. This

absence of a lower level of breaking load values was taken as an indication of the ability of the adsorbed organosilicon films to "heal" oxide defects and make the surface more homogeneous. It was thus discovered that treating oxidized silicon wafers with organosilicon compounds increases the uniformity of the physicochemical properties of the substrate surface. The technique of treating substrate surfaces with organosilicon compounds was thus recommended for use in improving the process characteristics of photoresists. Figure 1, table 1; references 9: 8 Russian, 1 Western.

#### Liquid-Phase Epitaxy of Isoperiodic $Ga_xIn_{1-x}As_ySb_{1-y}/GaSb$ Heterostructures

917M0172D Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 16 Jun 89) pp 684-688

[Article by A.N. Baranov, V.V. Kuznetsov, Yu.P. Yakovlev, A. Gyseynov, B.Ye. Dzhurtanov, I.A. Kachalova, M. Mirsagatov, and T.V. Cherneva, Leningrad Electrical Engineering Institute imeni V.I. Ulyanov]

UDC 548.25

[Abstract] The authors of the study reported herein examined the thermodynamic aspects of liquid-phase heteroepitaxy of  $Ga_xIn_{1-x}As_ySb_{1-y}/GaSb$  solid solutions of isoperiodic GaSb. The epitaxy was conducted in a horizontal quartz reactor in a flow of hydrogen that had been purified by diffusion through palladium. Non-doped InAs and GaSb served as the initial charge together with semiconductor-grade indium, gallium, and antimony totaling about 1.5 g. GaSb wafers oriented in the plane (100) and with an area of about 1 cm<sup>2</sup> served as the substrates. The solution-melt was homogenized at a temperature of 913 K for 2 hours, and the layers were deposited at 872 K over the course of 10 to 30 seconds. The thickness of the epitaxial films ranged from 2 to 3 μm. The experimental data obtained regarding the compositions of the resultant solid and liquid phases was used to correct the thermodynamic mixing functions on which the Ga-In-As-Sb phase diagram was computed. The authors succeed in obtaining isoperiodic  $Ga_xIn_{1-x}As_ySb_{1-y}/GaSb$  heterostructures where  $0.8 < x < 1.0$  and  $0.0 < y < 0.2$ . The liquid-phase epitaxy process used was substantiated within the framework of a thermodynamic model of simple solutions with consideration given to elastic deformations at the heteroboundary and mass transfer of the components in the liquid phase. Figures 3, tables 2; references 9: 4 Russian, 5 Western.

#### The Electrooptic Effect in the Ferroelectric $Sn_2P_2S_6$

917M0172E Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 14 Jun 89) pp 689-691

[Article by R.O. Blokh, Yu.M. Vysochanskiy, A.A. Grabar, A.V. Kityk, and V.Yu. Slivka, Uzhgorod State University]

UDC 537.226

[Abstract]  $Sn_2P_2S_6$  crystals undergo a unique ferroelectric phase transition of the second type with a change in  $P_{21/c} \rightarrow P_c$  symmetry at  $T_c = 339$  K. The authors of the study reported herein examined an induced electrooptic effect in  $Sn_2P_2S_6$  close to the phase transition. A quadratic electrooptic effect was possible in both phases of the crystals, whereas a linear electrooptic effect was forbidden in the centrosymmetric paraelectric phase. An electrooptic hysteresis loop was obtained in the ferroelectric phase, and a quadratic dependence of birefringence on the applied field was obtained in the paraelectric phase. The electrooptic coefficient and internal field were estimated. The discrepancy between the value found for the coercive field and the results of dielectric hysteresis studies were explained by the screening of the field inside the specimen by free charge carriers. The results obtained were seen as confirming the promise of using ferroelectric  $Sn_2P_2S_6$  crystals as an electrooptic material in the spectral range beyond the bounds of its photosensitivity. The research results also demonstrated that consideration must be given to screening effects when making measurements in electrostatic fields and when designing electrooptic elements. Figures 2; references 6: 5 Russian, 1 Western.

#### Anisotropy of the Optical Properties and Interlayer Interaction in Gallium Selenide

917M0172F Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 1 Jun 89) pp 692-695

[Article by V.V. Sobolev, V.Ye. Grachev, S.G. Kozlova, and V.Val. Sobolev, Applied Physics Institute, SSRM Academy of Sciences]

UDC 621.315

[Abstract] Layered materials have long been considered among the most promising for high-temperature superconductivity. After briefly reviewing the existing research on the properties of GaSe layered structures, the authors of this study report on their own studies of the reflection spectra of a GaSe crystal in the range from 1 to 5 eV at temperatures of 80 and 293 K for the polarization E || c. In the reflection spectra recorded at these two temperatures, the authors discovered (for the first time) a free exciton at 2.1 and around 10 in the interval from 2.2 to 2.5 eV that was attributed to interband transitions or metastable excitons. The authors computed complete complexes of fundamental optical functions in the case of the polarization E || c. They then proceeded to establish strong polarization, which directly confirmed the extensive anisotropy of interatomic interactions in GaSe. The similarity of the free excitons of GaSe and those of zinc diarsenides and diphosphides is also noted. Figures 3; references 14: 8 Russian, 6 Western.

### The Memory Properties of Silicon-Enriched Silicon Oxonitride

917M0172G Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 16 Jun 89) pp 733-736

[Article by B.I. Vasilyev, V.A. Gritsenko, and S.A. Kovtunenکو, Semiconductor Physics Institute, Siberian Department, USSR Academy of Sciences]

UDC 621.328

[Abstract] Amorphous silicon nitride is receiving a great deal of attention thanks to its ability to localize electrons and holes with an enormous retention time (about 10 years), i.e., thanks to its memory effect. In view of this fact, the authors of the study reported herein examined the memory properties of silicon oxonitride.  $\text{SiN}_x\text{O}_y$  layers were synthesized from the mixture  $\text{SiH}_4 + \text{NH}_3 + \text{H}_2\text{O}$ . A mixture of silane and hydrogen was used. The  $\text{SiH}_4$  volume concentration amounted to 0.2%. The volume concentration of ammonia in the hydrogen amounted to 60%. The  $\text{SiH}_4:\text{NH}_3$  ratio was varied by varying the flow rates. Hydrogen served as the carrier gas. The silicon oxonitride's memory properties were studied as a function of the ratio of the gas flows and the synthesis temperature. The simultaneous addition of excess silicon and oxygen to the silicon oxonitride was found to result in a reduction in the rate of charge leakage from the electrons and holes. It also caused an increase in the cyclic strength of the metal-nitride-oxide-semiconductor structure. The authors explained the reduction in charge leakage in the silicon oxonitride qualitatively within the framework of a polar model. They attribute the reduction to an increase in the energy of the localized states that is in turn the result of a decrease in the polar dielectric constant. The increase in cyclic strength is explained in terms of the replacement of the Si-H and N-H bonds by stronger Si-O bonds. Figures 3; references 6: 3 Russian, 3 Western.

### Distinctive Features of the Formation of a Superconductive Phase and Investigation of the Electronic Structure of Metal Oxides of Rare Earth Metals

917M0172H Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 19 Jan 89) pp 747-51

[Article by I.A. Massalimov, N.Yu. Arutyunov, N.Yu. Trashchakov, and Kh.T. Sharipov, Chemistry Institute, UzSSR Academy of Sciences]

UDC 537.312.62

[Abstract] In a continuation of the long line of studies on 123-type rare earth element superconductors, the authors of the study reported herein synthesized a

number of compounds of the type  $\text{RBa}_2\text{Cu}_3\text{O}_y$  (where R = Y, Nd, or Lu). These compounds were then subjected to x-ray analysis, thermography, and positron annihilation in order to shed further light on the dynamics of the formation of a superconductive phase and to determine the density of the pulsed distributions of electrons in the superconductive phase. Analysis-grade  $\text{Y}_2\text{O}_3$ ,  $\text{Nd}_2\text{O}_3$ ,  $\text{Lu}_2\text{O}_3$ ,  $\text{BaCO}_3$ ,  $\text{BaO}$ , and  $\text{CuO}$  were used to synthesize the following compounds:  $\text{RBa}_2\text{Cu}_3\text{O}_y$ ,  $\text{Nd}_1\text{Ba}_2\text{Cu}_3\text{O}_y$ , and  $\text{Y}_{0.5}\text{Lu}_{0.5}\text{Ba}_2\text{Cu}_3\text{O}_y$ . The compounds were synthesized in a two-stage process that is described in detail. Thermal analysis of specimens of the compounds synthesized indicated that superconductive material manufactured by using  $\text{BaCO}_3$  contains residues of unreacted barium carbonate. (This was confirmed by energy absorption at 815-818°C in the DTA curves that were associated with the phase transition to  $\text{BaCO}_3$ ). The observed endothermal effects occurring between 930 and 1,030°C were more intense for the mixture of components than for the superconductive material. This fact was interpreted to mean that two processes occur simultaneously in the former case, i.e., decomposition of the starting components and formation of a superconductive phase. Endothermal processes were observed to occur during the formation of the crystalline structure of the superconductive phase at temperatures between 980 and 1,100°C. Studies of the materials produced by positron annihilation revealed that the process of the annihilation of electron-positron pairs occurs primarily in  $\text{Cu}(1)\text{-O}(1)\text{-O}(4)$  chains located in layers in the microcrystals of the Y-Ba-Cu-O compounds studied. This behavior was attributed to the existence of an effective negative charge concentrated on the specified structural groups. Preliminary data led the authors to conclude that the reduction in the concentration of oxygen vacancies in the test specimens results in an expansion of the curve of the angular distributions of pairs of annihilation photons by more than 0.5 mrad and is accompanied by a significant decrease in the temperature of the transition to superconductivity ( $T_c$ ). Figures 3; references 11: 4 Russian, 7 Western.

### The Electron Structure and X-Ray Photoelectron Spectra of $\text{LnBa}_2\text{Cu}_3\text{O}_7$

917M0172I Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 5 Sep 89) pp 771-776

[Article by M.V. Ryzhkov, D.L. Novikov, Yu.F. Zhuravlev, M.V. Kuznetsov, and V.A. Gubanov, Chemistry Institute, Ural Department, USSR Academy of Sciences]

UDC 546.65:539.194

[Abstract] Because the electron structure of the compound  $\text{LnBa}_2\text{Cu}_3\text{O}_7$  has not yet been studied adequately, the authors of the study reported herein conducted experimental and theoretical studies of the electron states of  $\text{LnBa}_2\text{Cu}_3\text{O}_7$  with bond energies ranging from

35 to 0 eV. X-ray photoelectron spectroscopy and the cluster method of discrete variation were used for the studies, thus enabling the authors to model the electron state of both the group and excited states of the aforesaid system. Specimens in the form of ceramic tablets with diameters of 12 mm and thicknesses of 1 mm were manufactured according to a technology described elsewhere. The electron structure of the copper-oxygen sublattice in  $\text{LnBa}_2\text{Cu}_3\text{O}_7$  was modeled from computations for the following series of clusters:  $\text{Cu}_3\text{O}_{12}^{18-}$ ,  $\text{Cu}_6\text{O}_{21}^{30-}$ , and  $\text{Ba}_2\text{Cu}_{12}\text{O}_{3848-}$ . The  $\text{BaO}_{1018-}$  and  $\text{LnO}_8^{13-}$  (where  $\text{Ln} = \text{Y, Nd, Sm, Gd}$ ) clusters that included atoms of the metals and their closest neighbors were also examined separately. The authors demonstrated that the process of ionization of the Y, Cu, Ba, and O orbitals in  $\text{LnBa}_2\text{Cu}_3\text{O}_7$  occurs differently for the states of the valence range and for deeper levels. In the latter case, the electron vacancy, including that in the O2s orbitals, was found to be localized in one node. In the case of lanthanides, however, ionization of the 4f molecular orbitals was found to occur analogously to that of the delocalized O2p and Cu3d molecular orbitals despite the spatial compactness of the former. Moreover, interaction of the 4f orbitals with the valence band was observed to result in a marked decrease in the relaxation ship of the Ln5p levels to a level characteristic of valence-range states. Figures 4; references 17: 4 Russian, 13 Western.

#### Electron Structure and Chemical Bond in $\text{YBa}_2\text{M}_3\text{O}_7$ ( $\text{M} = \text{Cu, Ag, Au}$ )

917M0172J Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 5 Sep 89) pp 777-781

[Article by M.V. Ryzhkov, D.L. Novikov, and V.A. Gubanov, Chemistry Institute, Ural Department, USSR Academy of Sciences]

UDC 546.65:539.194

[Abstract] The primary objective of the study reported herein was to develop a theoretical model of the electron structure of  $\text{YBa}_2\text{M}_3\text{O}_7$  ( $\text{M} = \text{Ag, Au}$ ) and to then compare it (within the framework of a single methodology) to previously obtained data for the compound  $\text{YBa}_2\text{Cu}_3\text{O}_7$ . The authors based their modeling efforts on nonrelativistic ( $\text{Cu}_2\text{O}_{12}^{8-}$ ,  $\text{Ag}_3\text{O}_{12}^{18-}$ ) and relativistic ( $\text{Au}_3\text{O}_{12}^{18-}$ ) methods of discrete variation with correlation-exchange potential and expanded numeric atomic orbital bases. It was determined that partial or complete replacement of copper in  $\text{YBa}_2\text{M}_3\text{O}_7$  by Ag or Au with the same type of configuration should result in the formation of compounds with a similar electron structure, chemical bonding, and charge density distribution. This in turn confirmed the possibility of isostructural replacement of Cu by heavier metals with the configuration  $d^{10}s^1$ . The authors further concluded that the compounds  $\text{YBa}_2\text{Ag}_3\text{O}_7$  and  $\text{YBa}_2\text{Au}_3\text{O}_7$  contain two- and

one-dimensional structural elements and a partially filled band of the O2p-Mnd type in the upper part of the valence band. It was thus concluded that both hypothetical systems may be high-temperature superconductors. Because of the reduced state density at the Fermi level in silver-containing compounds as opposed to in copper-containing compounds, the authors stated that they would expect the temperature of the transition to superconductivity ( $T_c$ ) to be lower for  $\text{YBa}_2\text{Ag}_3\text{O}_7$  than for  $\text{YBa}_2\text{Au}_3\text{O}_7$ . They further speculated that the nature of conduction in a gold-containing compound may differ from that of the compound  $\text{YBa}_2\text{Cu}_3\text{O}_7$ . Figures 2, table 1; references 19: 2 Russian, 17 Western.

#### The Possibility of Replacing Barium by Calcium (Strontium) in Aluminates

917M0172K Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 16 Jun 89) pp 782-785

[Article by L.N. Lykova, M.V. Paromova, Z.Ya. Kulikova, O.N. Rozanova, O.Yu. Maslennikov, and V.V. Gruzdeva, Moscow State University imeni M.V. Lomonosov]

UDC 546.431.621

[Abstract] The authors of the study reported herein examined the possibility of replacing barium by calcium (strontium) in aluminates of the compositions  $\text{Ba}_4\text{Al}_2\text{O}_7$  and  $\text{Ba}_7\text{Al}_2\text{O}_{10}$  and to refine the bounds of the existence of solid solutions based on these compounds. For their experiments, the authors synthesized barium-calcium (strontium) specimens with the general formulas  $\text{Ba}_{4-x}\text{Ca}_x(\text{Sr})\text{Al}_2\text{O}_7$  ( $x = 0$  to 2) and  $\text{Ba}_{7-x}\text{Ca}_x(\text{Sr})\text{Al}_x\text{O}_{10}$  ( $x = 0$  to 1) in accordance with the solid-phase technique. X-ray analysis of the specimens with the general composition  $\text{Ba}_{4-x}\text{Ca}_x(\text{Sr})\text{Al}_2\text{O}_7$  revealed that the range of solid solutions based on  $\text{Ba}_4\text{Al}_2\text{O}_7$  is rather wide and exists all the way to the composition  $\text{Ba}_{3.4}\text{Ca}_{.6}\text{Al}_2\text{O}_7$ . In the composition interval from  $\text{Ba}_{3.4}\text{Ca}_{.6}\text{Al}_2\text{O}_7$ , the specimens represented two-phase mixtures of a solid solution based on  $\text{Ba}_4\text{Al}_2\text{O}_7$  and the compound  $\text{Ba}_3\text{CaAl}_2\text{O}_7$ . When the calcium content in  $\text{Ba}_{4-x}\text{Ca}_x(\text{Sr})\text{Al}_2\text{O}_7$  was increased so that  $x$  was between 1.1 and 2.0, the composition of the test specimens was found to correspond to the three-phase range (solid solutions based on  $\text{Ba}_3\text{CaAl}_2\text{O}_7$ ,  $\text{Ba}_2\text{Al}_2\text{O}_6$ , and  $\text{CaO}$ ). Studies of the section  $\text{Ba}_4\text{Al}_2\text{O}_7$ - $\text{Sr}_4\text{Al}_2\text{O}_7$  in the system  $\text{BaO-SrO-Al}_2\text{O}_3$  at  $1,200^\circ\text{C}$  revealed that the range of a solid solution based on  $\text{Ba}_4\text{Al}_2\text{O}_7$  is rather narrow and includes  $\text{Ba}_{3.8}\text{Sr}_{0.2}\text{Al}_2\text{O}_7$ . Contrary to data that have been published in several other sources, the authors of the study reported herein found that  $\text{Ba}_7\text{Al}_2\text{O}_{10}$  does indeed exist at temperatures above  $1,200^\circ\text{C}$ . For that reason, they proceeded to investigate the possibility of replacing barium by calcium (strontium) in that compound as well. They concluded that barium-calcium aluminates based on the said compound have an elevated hygroscopicity and therefore are

not feasible for use in manufacturing porous metal cathodes. The authors did, however, determine that their new compound  $Ba_3CaAl_2O_7$  is promising for use in thermionic materials. Figure 1, table 1; references 8: 3 Russian, 5 Western.

#### Investigation of Dye Centers in $YScO_3$ and $LuScO_3$ Monocrystals

917M0172L Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 16 Jun 89) pp 795-799

[Article by A. Abdurazakov, V.A. Antonov, P.A. Arsenyev, Kh.S. Bagdasarov, and A.M. Kevorkov, Moscow Power Institute]

UDC 535.343.2:341.67

[Abstract] The authors of the study reported herein examined the point defects of the monocrystalline structures of  $YScO_3$  and  $LuScO_3$  resulting from dyeing the crystals under the effect of exciting radiation. The starting compounds were produced from yttrium, lutetium, and scandium by the conventional solid-phase synthesis techniques.  $YScO_3$  and  $LuScO_3$  monocrystals were grown by the method of oriented crystallization in cylindrical molybdenum containers in an inert-reducing atmosphere at a rate of 1.5 mm/h. The crystals were subjected to  $\gamma$ -irradiation and then dyed. Spectroscopy studies performed on the crystals revealed that they reached saturation at irradiation doses of  $6 \times 10^6$  R. The experiments conducted pointed to the formation of a bulk charge (electrons) during the irradiation of the test crystals and that the rate of the process of spreading depends on the specimens' temperature. It was determined that these electrons may be captured by the crystals' structural defects (resulting in the formation of dye centers) and may also be recombined in them (which is manifested as an increase or decrease in the intensity of the respective additional absorption bands). The thermal breakdown of these defects was found to occur at high temperatures. The effects of x-irradiation on heat-stimulated processes in  $YScO_3$  and  $LuScO_3$  crystals were also examined. The authors also discovered that adding  $Nd^{3+}$  ions to  $LuScO_3$  and  $YScO_3$  crystals increases their radiation resistance. This effect is apparently due to the fact that the  $Nd^{3+}$  ions create additional recombination channels, which significantly reduces the concentration of dye centers in both types of crystals. Figures 3, table 1; references 6 (Russian).

#### The Electron Structure and Electric Conduction of Rutile-Like Solid Solutions of $Ru_{1-x}M_xO_2$ (M = V, Nb, W)

917M0172M Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 16 Jun 89) pp 832-836

[Article by Yu.S. Shorikov, L.N. Mazalov, E.A. Kravtsova, G.K. Parygina, and Ye.A. Rummyantseva, State

Central Scientific Research, Planning, and Design Institute of Precious Metals and Diamonds]

UDC 669.236+539.264+537.531+535.3

[Abstract] The authors of the study reported herein examined the electron structure and electric conduction of rutile-like solid solutions of  $Ru_{1-x}M_xO_2$  (M = V, Nb, W) that were obtained by heat-treating the compositions  $RuO_2 + V_2O_5$ ,  $RuO_2 + t-Nb_2O_5$ , and  $RuO_2 + WO_3$  in air at 1,473, 1,573 and 1,523 K for 1, 30, and 10 hours, respectively. The said specimens were then subjected to x-ray fluorescence analysis. The authors discovered that in the compounds  $RuO_2$  and  $Ru_{1-x}M_xO_2$ , the d-bands of the cationic sublattice formed by the  $2t_{2g}$ -orbitals are located close to the Fermi level and that the energy levels of the metal-oxygen bond are localized in the lower part of the valence band. They further discovered that when  $RuO_2$  is alloyed to a level of  $x = 0.05-0.1$ , the energy of the  $2t_{2g}$  band is intensified because of the intercationic Ru-M interaction. The density of the 4d "metal" states of the Ru in the said compound is, on the other hand, found to decrease. This in turn results in a sharp increase in the value of  $\rho$  (in this case,  $W > Nb > V$ ). As the content of M is increased, the position of the  $2t_{2g}$  levels stabilizes. Because of the decrease in the 2p-density of O at the Ru-O  $\pi$ -bond, however, the respective 4d-electrons are delocalized where the M-O  $\pi$ -band appears, and the value of  $\rho$  decreases somewhat. Figure 1, tables 2; references 7: 5 Russian, 2 Western.

#### The Phase Composition and Properties of Sodium-Doped $YBa_2Cu_3O_{7-\delta}$ Ceramic

917M0172N Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 23 Jun 89) pp 846-848

[Article by G.N. Novitskaya K.P. Danilchenko, A.G. Popov, V.M. Pan, and A.N. Antishko, General and Inorganic Chemistry Institute, UkSSR Academy of Sciences, and Metal Physics Institute, UkSSR Academy of Sciences]

UDC 546.65:539.194

[Abstract] The authors of the study reported herein examined the effect of sodium on the phase composition and properties of specimens of  $(Y_{1-x}Na_{x,3x})Ba_2Cu_3O_{7-\delta}$ . Two types of specimens were prepared. In the first type of specimen yttrium and sodium were given consideration. No consideration was given to valence in the second group of specimens. Ten different specimen compositions were synthesized. Experiments conducted on the specimens established that substituting sodium for part of the yttrium in  $YBa_2Cu_3O_{7-\delta}$  does indeed affect the phase composition and properties of this high-temperature superconducting ceramic. When the excess sodium amounts to 0.1 mole (in relation to the stoichiometric proportions [in other words, 0.3% by

weight]), the high-temperature superconductor phase  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  is preserved, the temperatures required to synthesize and sinter the ceramic increase, and the temperature of the transition to superconductivity ( $T_c$ ) decreases. Large quantities of sodium were not only found to disturb phase homogeneity and increase the ceramic's synthesis and sintering temperatures but were also found to increase its specific resistance and thus significantly intensify its semiconducting component. The authors found that adding sodium in the form of NaF not only had all of the aforementioned consequences but also resulted an increase in the ceramic's specific resistance by 5 to 6 orders of magnitude. Table 1, references 9 (Russian).

### Anisotropy of the Absorption of $\gamma$ -Radiation by Monocrystals of Layered Semiconductors

917M01720 Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 26 Mar 89) pp 856-857

[Article by G.D. Guseynov, R.A. Akopyan, V.M. Zhuravlev, E.G. Abdullayev, and S.Kh. Umarov, Physics Institute, AzSSR Academy of Sciences]

UDC 537.311.33

[Abstract] Type  $A^{III}B^{VI}$  semiconductors and their complex analogues exhibit a high sensitivity to x- and  $\gamma$ -radiation and are thus suitable for use in radiation filters and diffraction analyzers or, possibly, radiation detectors. Because these applications require clarification of the relationship between these crystals' absorption and their orientation and chemical composition, the authors of this concise communication undertook just such a study. They examined specimens of crystals grown by the Bridgman method. The specimens were subjected to  $\gamma$ -irradiation from  $^{60}\text{Co}$  and  $^{137}\text{Cs}$  sources. Their coefficients of linear absorption were then determined by using an expression provided by the authors. The results are detailed in table form. The anticipated coincidence of the calculated and experimentally obtained values was not obtained. This fact was attributed to both computational and experimentation errors. The authors further concluded that the experimentally observed anisotropy was in all likelihood due to the asymmetry of chemical bonding that is inherent to all crystals of the given types of compounds and thus to the differing reticular (atomic) densities in different crystallographic directions. Figure 1, table 1; references 7 (Russian).

### The Formation Enthalpy of $\text{YBa}_2\text{Cu}_3\text{O}_7$ : A Peroxide Alternative

917M0172P Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 27 No 4, Apr 91 (manuscript received 26 Mar 90) p 872

[Article by L.A. Reznitskiy; Moscow State University imeni M.V. Lomonosov]

UDC 536.63:541.11

[Abstract] Most publications dealing with the crystalline structure and thermodynamic properties of the high-temperature oxide superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_7$  speak of the presence of one trivalent copper ion per formula unit. The authors of this concise report cite one work proposing that under normal synthesis conditions, all of the copper ions are bivalent. Proceeding from this assumption, the authors of the present communication develop a new estimate of the formation enthalpy of  $\text{YBa}_2\text{Cu}_3\text{O}_7$ . This new estimate is developed by proceeding from the assumption of the presence of 0.5 mole  $\text{BaO}_2$  and bivalent copper but using the same polyhedral description of the aforesaid superconductor's structure that has been assumed in articles subscribing to the trivalent copper theory. The authors arrive at a value of  $-2,714$  kJ/mol, which is in fairly good agreement with the experimentally determined value of  $-(2,719 \pm 18)$  kJ/mol and also fairly close to the value calculated under the assumption of trivalent copper ions, i.e.,  $-(2,699 \pm 8)$  kJ/mol. References 4: 3 Russian, 1 Western.

### Nuclear Spin Echo in Thin Cobalt Films Due to Radio-Frequency Pulses With Noise and Harmonic Filling

917M0173A Moscow VESTNIK LENINGRADSKOGO UNIVERSITETA: FIZIKA, KHIMIYA, MEKHANIKA in Russian No 1, Feb 91 (manuscript received 5 Jun 90) pp 86-88

[Article by Ye.G. Apushkinskiy and V.V. Moskalev]

UDC 539.143.43

[Abstract] Research has demonstrated that different types of radio-frequency pulses induce very different echo signals. The authors of the study reported herein conducted a series of experiments to determine the effect of rectangular radio-frequency pulses with harmonic and noise carriers in thin cobalt films. The films were manufactured in accordance with a method described elsewhere, and the nuclear echo was excited by using the conventional two- and three-pulse method. An S4-74 spectrum analyzer was used to analyze the pulses' spectra. One of the pulses in each sequence was rather short and had a harmonic carrier, while the others had noise carriers. Both the experiments and computations performed by the authors revealed that increasing the duration of the aforesaid pulses without reducing their amplitude results in a very intensive effect on a narrow region of the spin bursts. This in turn distorts the echo signals. It is further discovered that this effect may be avoided by reducing the amplitude of the long pulse or producing it with a noise carrier. Figures 2; references 4 (Russian).

**Thermal Studies of the High-Temperature Transformations of the Crystalline Structure of  $Y_1Ba_2Cu_3O_y$**

917M0173B Moscow VESTNIK LENINGRADSKOGO UNIVERSITETA: FIZIKA, KHIMIYA, MEKhanIKA in Russian No 1, Feb 91 (manuscript received) pp 99

[Article by T.V. Belopolskaya, G.I. Tsereteli, S.K. Filatov, V.V. Semin, O.I. Smirnova, and T.V. Gracheva]

UDC 537.226:537.313.62

[Abstract] The authors of the study reported herein conducted comprehensive research on the high-temperature transformations of the crystalline structure of both the rhombic and tetragonal phases of  $Y_1Ba_2Cu_3O_y$  by the methods of differential thermal analysis, x-ray structural analysis, and thermal desorption mass spectroscopy. Rhombic phase specimens ( $a = 0.836$  (1) nm,  $b = 0.3887$  (2) nm,  $c = 1.1669$  (6) nm and various tetragonal phase specimens (the most detailed having parameters of  $a = 0.3862$  (1) nm,  $c = 1.1746$  (1) nm) in which no extraneous impurities were detectable were used for the studies. The rhombic specimens were produced by using the conventional ceramic technology, and the tetragonal specimens were produced by hot pressing in a vacuum. The research conducted demonstrates that all exothermal and endothermal processes occurring as specimens of the aforesaid ceramics are heated from room temperatures up to the melting point may be attributed to the absorption or release of oxygen. The exothermal effect noted from 503 to 873 K for various tetragonal phases was determined to correspond to oxygen absorption with the transition to the rhombic phase. Heating at temperatures from 783 to 1,043 K was found to induce oxygen release and stabilize the tetragonal phase in both specimens that were originally rhombic and specimens that were initially tetragonal. The endothermal effect observed by the researchers in a narrow range of temperatures around 1,073 K was attributed to oxygen release. It was further speculated that this effect corresponds to a weak structural transformation that cannot be detected roentgenographically. Figures 4, table 1; references 8: 3 Russian, 5 Western.

**A Magnetochemical Study of  $YCaCr_xAl_{1-x}O_4$  Solid Solutions**

917M0173C Moscow VESTNIK LENINGRADSKOGO UNIVERSITETA: FIZIKA, KHIMIYA, MEKhanIKA in Russian No 1, Feb 91 (manuscript received) 25 Jun 90) pp 108-110

[Article by I.A. Zvereva, N.F. Anashkina, and N.V. Chezhina]

UDC 54-165:537.621

[Abstract] In a continuation of research on the effect of rare earth elements on the magnetic properties of complex oxides and their solid solutions, the authors of the

study reported herein examined the magnetic and structural characteristics of  $YCaCr_xAl_{1-x}O_4$ . They synthesized  $YCaCr_xAl_{1-x}O_4$  ( $0.01 \leq x \leq 0.15$ ) in accordance with the ceramic method by roasting in air at a temperature of 1,710 K for 30 hours. X-ray analysis was used to control the solutions' homogeneity, and the chromium content was determined by the photocolometric method. Magnetic susceptibility was measured by the Faraday method in the temperature interval from 77 to 420 K with a measurement error not exceeding 1%. The experiments revealed that replacing lanthanum by yttrium results in an increase in magnetic susceptibility in systems with layered and perovskite structures alike. The curve of the dependence  $\chi_{Cr}^{vapor}(x)$  in yttrium-containing systems was found to have a sharper drop in the range of concentrations studied. In lanthanum-containing systems, on the other hand, magnetic susceptibility was found to first fall sharply as the concentration increased but to then decrease more smoothly. The authors attributed this behavior to the fact that, in view of the different polarizing effect of rare earth elements on oxygen atoms, the aggregation of paramagnetic chromium atoms is stronger in lanthanum-containing systems. Consequently, large paramagnetic groupings whose susceptibility is not so different from that of single chromium atoms may form in lanthanum-containing systems at lower concentrations than is possible in yttrium-containing systems. It is this fact that the authors determine to be responsible for the more even course of the curve  $\chi_{Cr}^{vapor}(x)$ . Figures 2; references 6: 4 Russian, 2 Western.

**The Magnetic Susceptibility of  $YCaNi_xAl_{1-x}O_4$  Solid Solutions**

917M0173D Moscow VESTNIK LENINGRADSKOGO UNIVERSITETA: FIZIKA, KHIMIYA, MEKhanIKA in Russian No 1, Feb 91 (manuscript received) 29 Jun 90) pp 110-112

[Article by N.V. Chezhina, N.V. Savelyeva, and G.N. Yachnaya]

UDC 54-165:537.621

[Abstract] Others have studied the magnetic behavior of nickel (III) atoms in  $LaSrNi_xAl_{1-x}O_4$  and have discovered that the spin state of the nickel atoms exerts a significant effect on the sublattice of heavy metals. In an effort to clarify this effect, the authors of the study reported herein examined the magnetic susceptibility of solid solutions with the same  $K_2NiF_4$ -type structure but with lanthanum and strontium replaced by yttrium and calcium. In other words, they examined the magnetic susceptibility of  $YCaNi_xAl_{1-x}O_4$ . Solid solutions of  $YCaNi_xAl_{1-x}O_4$  ( $0.005 \leq x \leq 0.10$ ) were synthesized by the ceramic method from yttrium, aluminum, and nickel (II) oxides and calcium carbonate. The specimens were roasted for 30 hours at a temperature of 1,670 K in air. Magnetic susceptibility was measured by the Faraday method in the temperature range from 77 to 400 K with

a precision to 1%. The studies demonstrated that the nickel (III) atoms exist in two ground states (i.e., low-spin and high-spin) even at low concentrations. The authors attributed this fact to the nonequivalence of the crystalline fields of the Y and Ca atoms. It was thus discovered that in the case of  $\text{YCaNi}_x\text{Al}_{1-x}\text{O}_4$  solid solutions, the maximum in the isothermic line of the paramagnetic component of magnetic susceptibility is shifted to the left as compared with that for  $\text{LaSr}_x\text{Al}_{1-x}\text{O}_4$  solid solutions. Figure 1, tables 2; references 2 (Russian).

### The Synthesis and Magnetic Susceptibility of $\text{La}_{1.85}\text{Sr}_{0.15}\text{CuO}_4$ - $\text{LaSrAlO}_4$ Solid Solutions

917M0173E Moscow VESTNIK LENINGRADSKOGO UNIVERSITETA: FIZIKA, KHIMIYA, MEKHANIKA in Russian No 1, Feb 91 (manuscript received 15 Jul 90) pp 112-114

[Article by N.P. Bobrysheva and Ye.A. Shchupunov]

UDC 669.018:537.312.62

[Abstract] The authors of the study reported herein attempted to examine the state of copper in superconductors of the La-Ba(Sr)-Cu-O type by using the magnetic dilution technique. As a test subject they selected  $\text{La}_{1.85}\text{Sr}_{0.15}\text{CuO}_4$ - $\text{LaSrAlO}_4$  solid solutions, which is a complex  $\text{LaSrAlO}_4$  oxide having a  $\text{K}_2\text{NiF}_4$ -type tetragonally distorted lattice.  $\text{La}_{1.85}\text{Sr}_{0.15}\text{CuO}_4$ - $\text{LaSrAlO}_4$  solid solutions were synthesized by two methods—the ceramic method and nitrate decomposition. The magnetic susceptibility measurements taken reveals that the copper present in the aforesaid solid solutions exists in two oxidized states, i.e., Cu (II) and Cu (III). The latter is demonstrated to be in a low-spin state. References 2 (Russian).

### Obtaining $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ Yttrium-Barium Cuprate From Solutions

917M0174A Moscow UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 57 No 6, Jun 91 (manuscript received 30 May 90) pp 586-589

[Article by G.N. Novitskaya, V.S. Flis, V.M. Pan, A.A. Flis, S.V. Polyanetskaya, and K.P. Danilchenko, General and Inorganic Chemistry Institute, UkSSR Academy of Sciences, Kiev, Metallophysics Institute, UkSSR Academy of Sciences, Kiev, and Materials Science Problems Institute, UkSSR Academy of Sciences, Kiev]

UDC 538.945

[Abstract] In view of the promise of the technique of producing  $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$  by coprecipitation from solutions (i.e., the chemical method of synthesis), the authors of the study reported herein examined the compounds NaOH,  $\text{NaH}_2\text{OH}$ ,  $(\text{NH}_4)_2\text{CO}_3$ , and  $\text{H}_2\text{C}_2\text{O}_4$ , which are used as precipitating agents in the production of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$  from solutions. Because coprecipitation of yttrium, barium, and copper in the form of hydroxides is

impossible owing to the significant differences in the chemical properties of yttrium and copper and the solubility of  $\text{Ba}(\text{OH})_2$ , the authors studied the precipitating agents KOH and  $\text{Ba}(\text{OH})_2$ .  $\text{Ba}(\text{OH})_2$  and KOH permitted the coprecipitation of yttrium and copper in the form of the hydroxides  $\text{Y}(\text{OH})_3$  and  $\text{Cu}(\text{OH})_2$ . A pH of 10 was found to be optimal for precipitation of copper in the form  $\text{Cu}(\text{OH})_2$ , and a pH of 7 was found to be optimal for the precipitation of yttrium in the form  $\text{Y}(\text{OH})_3$ . Barium was added to the washed precipitate in the form of an aqueous solution of the hydroxide  $\text{Ba}(\text{OH})_2$ . Elemental analysis of the  $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$  powder thus produced indicated that its composition corresponds to the specified stoichiometry. It was found to undergo the superconductive transition at temperatures between 89 and 94 K. The powder proved to be active to sintering, and the ceramic had a sintering temperature 40 to 50°C below that of powder produced via the conventional ceramic technology (960°C;  $\tau$ , 12 to 20 hours). The powder was formed into tablets 10 mm in diameter and 3 to 4 mm thick with a density of 5.5 to 5.7 g/cm<sup>3</sup>. Measurements of the relative electric resistance of the powders produced indicated that using the precipitating agent  $\text{Ba}(\text{OH})_2$  results in a change in conduction from the metal type to the semiconductor type;  $\text{Ba}(\text{OH})_2$  was thus determined to require further modification. KOH, on the other hand, results in a single-phase high-temperature superconductor with a critical temperature of 89 to 93 K, a critical temperature differential of 4 to 5 K, and a 100% transition to a superconductive state. Figures 2; references 6 (Russian).

### Production and Optical Properties of Monocrystals of Solid Solutions of the System $\text{AgGaS}_2$ - $\text{AgGaSe}_2$

917M0174B Moscow UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 57 No 6, Jun 91 (manuscript received 21 Jun 90) pp 589

[Article by I.M. Stoyka, L.M. Suslikov, L.S. Gaza, and Z.P. Gadmashi, Uzhgorod University]

UDC 548.56+546.571+535.345.6

[Abstract] The crystals  $\text{AgGaS}_2$  and  $\text{AgGaSe}_2$  have high nonlinear optical susceptibility coefficients and have recently aroused interest in the context of the creation of a new type of wide-angle selective light filters based on the effect of the interaction of orthogonally polarized modes. In view of this promising application, the authors of the study reported herein developed a technology for producing large  $\text{AgGa}(\text{S}_{1-x}\text{Se}_x)_2$  monocrystals. The new technology calls for using type GA 99.9997 gallium, ultrapure 16-5 sulfur, ultrapure 21-4 silver, and Sr 99.99 silver. The sulfur and silver are subjected to additional purification by vacuum distillation and band melting, respectively. The charge is synthesized by a two-temperature method from stoichiometric quantities of the elementary components in double quartz ampules evacuated to  $10^{-4}$ . The charge-containing ampules are

placed in a furnace and turned continuously at a speed of 0.26 to 0.35<sup>-1</sup> until the furnace reaches a working temperature of 1,275 to 1,375 K so that one-third of the charge is at a temperature of 360-400 K and two-thirds is at the maximum temperature. The ampules are held at this temperature for 20 to 40 hours and then moved into the hot zone at a speed of 2 to 5 mm/h. The synthesis process is conducted at maximal temperature for 12 to 16 hours. The ampules are then cooled to room temperature after the furnace has been shut off. The resultant charge is used to grow monocrystals in graphitized ampules in accordance with the Bridgman method. Among other things, studies performed on the crystals produced revealed that as the percentage of selenium in the crystals increases, the "isotropic" point shifts monotonically to the long-wave range of the spectrum. The technology developed results in large crystals of high uniformity and good optical quality that are suitable for use in nonlinear optics devices and filtration of optical radiation. Figures 3, table 1; references 7: 4 Russian, 3 Western.

#### The Physical Chemistry of Materials Based on CdSb-NiSb (CoSb, MnSb) Eutectics Grown by Oriented Crystallization

917M0174C Moscow UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 57 No 6, Jun 91  
(manuscript received 23 May 90) pp 597-601

[Article by A.A. Ashcheulov, D.P. Belotskiy, V.A. Grebenshchikov, and S.M. Kulikovskaya, Chernovtsy University]

UDC 546:621.362.002

[Abstract] The authors of the study reported herein used the methods of differential thermal and x-ray microanalysis to examine the physicochemical properties of materials based on the system CdSb-NiSb (CoSb, MnSb). The study alloys were synthesized in quartz, graphitized, hydrogen-filled ampules over the course of several days. The synthesis process included heating the specimens in a rotary furnace at temperatures in the range from 865 to 1,450 K and then cooling them at a rate of 120 to 150 K/h. The differential thermal and microstructural analyses performed revealed that the CdSb-NiSb, CdSb-CoSb, and CdSb-MnSb sections studied are quasi-binary. Their phase diagrams were found to belong to the eutectic class and are shifted along their composition axes to the side of the low-alloy component CdSb. Two other effects besides those corresponding to a eutectic and liquidus were noted in the said phase diagrams. The first corresponded to a magnetic transformation at 1,223 K, and the second corresponded to a phase transition at

823 K. Both effects occur in the phase diagram of the system Co-Sb from the side of the Co-CoSb. The study alloys were demonstrated to be rod-type alloys with regularly arranged round rods. The CdSb-MnSb eutectics were found to have a lengthwise band structure manifested in the form of a waveform disturbance in the temperature field at the crystallization front that leads to a periodic disappearance of the latter's stability. These and other study results presented were used in creating a commercial technology for producing temperature stability-optimized material for anisotropic thermocouples based on CdSb-MeSb oriented-crystallization eutectics, where Me = Ni, Co, and Mn. These materials may be used to create an entire series of new portable instruments and devices with a measurement precision of at least 1.5. Figures 4, tables 3; references 6 (Russian).

#### The Reaction of Benzene With CrO<sub>2</sub>Cl<sub>2</sub>-Modified Silica Surface

917M0174D Moscow UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 57 No 6, Jun 91  
(manuscript received 21 Jun 90) pp 608-610, 672

[Article by N.V. Borisenko, Yu.V. Plyuto, and A.A. Chuyko, Surface Chemistry Institute, UkSSR Academy of Sciences, Kiev]

UDC 541.183

[Abstract] Redox transformations involving chromium (VI) oxo anions in the surface layer of disperse silica have been the subject of intensive research in connection with the extensive use of the system CrO<sub>2</sub> in catalytic hydrogenation, oxidation, and polymerization reactions. One problem with using chromium (VI) in such reactions is that the clearly pronounced oxidizing properties of chromium (VI) oxo anions sharply restrict the set of electron-donor molecules-bands. Benzene, which is characterized by a rather high ionization potential (9.25 eV), is promising for use in studying the processes of the complex formation of chromium (VI) oxo anions. For this reason, the authors of the study reported herein used spectral methods to examine the nature of the reaction of benzene with chromium (VI) oxo anions on the surface of disperse silica. They reacted CrO<sub>2</sub>Cl<sub>2</sub> with Aerosil that had been dehydrated at 973 K ( $S_{\text{spec}} = 300 \text{ m}^2/\text{g}$ ). The reaction was conducted at 420 K for 1 hour. The experiment established that reacting benzene with a chlorochromyl group-carrying silica surface results in the formation of donor-acceptor complexes resulting from the transfer of electron density from the benzene molecule to the chromium atom of the grafted group. The formation of these complexes was found to be reversible and to not entail the oxidation of benzene. Figures 3; references 14: 6 Russian, 8 Western.

**The Catalysis Mechanism of Arbuzov's Reaction by Complex Transition Metal Compounds. 6. A Study of the Role of Palladium (II) Chloride in the Phosphorylation of Aryl Halides**

917M0162A Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript  
received 9 Aug 88) pp 370-373

[Article by V.V. Sentemov, Ye.A. Krasilnikova, and I.V. Berdnik, Izhevsk Agricultural Institute and Kazan Chemical Technology Institute imeni S.M. Kirov]

UDC 546.98+541.128.1

[Abstract] The authors of the study reported herein examined the role of palladium (II) chloride in the phosphorylation of aryl halides. They extracted the NMR spectra of  $^{31}\text{P}$  on a Bruker WP-80 spectrometer with a working frequency of 32.4 MHz. The spectra of the reaction mixtures were taken in a mode of protracted accumulation (up to 40 minutes). Chemical shifts of the phosphorus nuclei relative to 85%  $\text{H}_3\text{PO}_4$  were indicated. The IR spectra of the complexes in the long-wave range were taken on a Perkin-Elmer 577 spectrometer. All systems subjected to spectroscopic studies were prepared directly before measurement of their physical parameters. The studies revealed that the reaction of nonactivated aryl halides with trialkyl phosphites in the presence of catalytic quantities of palladium (II) chloride is a complex multistep process that includes the formation of the phosphorus (III)-containing palladium coordination compounds Pd (II), Pd (I), and Pd (0). Palladium coordination compounds in low degrees of oxidation formed as a result of the reaction occurring when the excess ethers of P (III) acids react with palladium (II) chloride are determined to be the likely activators of the reaction under study. References 18: 15 Russian, 3 Western.

**Catalysis Mechanism of Arbuzov's Reaction by Complex Transition Metal Compounds. 7. Catalysis of Arbuzov's Reaction by Organophosphorus Palladium Complexes**

917M0162B Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript  
received 18 Nov 88) pp 374-376

[Article by V.V. Sentemov, Ye.A. Krasilnikova, and I.V. Berdnik, Izhevsk Agricultural Institute and Kazan Chemical Technology Institute imeni S.M. Kirov]

UDC 546.98

[Abstract] The authors of the study reported herein examined the catalysis of Arbuzov's reaction by organophosphorus palladium complexes. The various prospective catalysts tested were synthesized according to methods published elsewhere. In each of the experiments conducted, aryl halide and the test catalyst were loaded into a reactor, and argon or nitrogen was passed through.

The reactor was then placed into a bath that was heated to 180°C. After 10 to 15 minutes, 1 mole of P (III) compound in relation to the aryl halide was added. The mixture was then held at the given temperature for 5 minutes. If no reaction occurred, the temperature was raised in successive 5°C increments. After the reaction began, drops of P (III) compounds were added at a rate of 1 drop/s. The reaction mixture was kept at a constant temperature as alkyl halide was released. A Bruker WP-80 spectrometer was used to record the NMR spectra, and a UR-20 spectrometer was used for the IR spectra. The following compounds were found to be catalysts of Arbuzov's reaction by nonactivated aryl halides with organic derivatives of P (III):  $\text{PdX}_2$ ,  $\text{K}_2\text{PdX}_4$ ,  $\text{Pd}[(\text{EtO})_3\text{P}]_2\text{X}_2$ ,  $\text{Pd}[(i\text{-PrO})_3\text{P}]_2\text{X}_2$ ,  $(\text{PdPPh}_3\text{Cl})_2\text{CO}$ ,  $(\text{PdPPh}_3\text{OAc})_2\text{CO}$ ,  $\text{Pd}(\text{PPh}_3)_3$ , and  $\text{Pd}(\text{Ph}_2\text{PC}_6\text{H}_4\text{F})_2\text{PPh}_3$ . The complexes  $\text{Pd}(\text{O})\text{-Pd}(\text{PPh}_3)_3$  and  $\text{Pd}(\text{Ph}_2\text{PC}_6\text{H}_4\text{F})_2\text{PPh}_3$  were found to manifest the greatest activity. References 18: 15 Russian, 3 Western.

**Catalysis Mechanism of Arbuzov's Reaction by Complex Transition Metal Compounds. 8. The Electrochemical Behavior of Complexes of Nickel (II) With Derivatives of P (III) Acids in Acetone**

917M0162C Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript  
received 23 Nov 88) pp 376-380

[Article by I.V. Postnova and V.V. Sentemov, Izhevsk Agricultural Institute]

UDC 541.13:541.49:541.128:546.74.547.557

[Abstract] The authors of the study reported herein studied the electrochemical behavior of complexes of nickel (II) with derivatives of P (III) acids in acetone. A PO-5122A oscillopolarograph and PPT-1 polarograph were used for the electrochemical studies. A saturated calomel electrode served as the standard electrode, and a dropping mercury electrode with a drop period of 10.8 seconds that was used in a forced-drop separation mode served as the working electrode. Settled mercury served as an auxiliary electrode. Carefully purified argon was used to free the polarographed solution of oxygen. The solutions were subjected to polarography at 18°C. Freshly prepared solutions (cleaned by a method published elsewhere) were used in the polarographic studies.  $\text{NaClO}_4$  and  $(\text{C}_2\text{H}_5)_4\text{NClO}_4$  were used as background salts, and acetone and benzene served as solvents. The background salts were twice recrystallized from aqueous and alcohol solutions. Oxygen was first removed from the solution of nickel (II) perchlorate in acetone by blowing argon in for 30 minutes, then adding phosphite, and again flowing argon through the reactor for 5 minutes. Complexes of nickel (II) with  $\text{P}(\text{OEt})_3$ ,  $\text{EtP}(\text{OEt})_2$ , and  $(\text{CH}_2\text{O})_2\text{POEt}$  were found to form in the presence of two- to fivefold excesses of ligands and with 100- to 400-fold excesses of silylphosphites. All of the complex nickel (II) compounds studied with derivatives of P (III)

acids were reduced to nickel (O) compounds by single-stage two-electron transfer. Figure 1, table 1; references 16: 13 Russian, 3 Western.

#### Hydrophosphoryldibenzo-14-Crown-5 and Its Transformations

917M0162D Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript  
received 18 May 89) pp 380-384

[Article by R.I. Yurchenko, V.G. Yurchenko, T.I. Klepa, and A.M. Pinchuk, Institute of Organic Chemistry, UkSSR Academy of Sciences, Kiev]

UDC 546.183+547.639

[Abstract] The crown ether hydrophosphoryldibenzo-14-crown-5 opens up a whole new approach to synthesizing phosphorylated macrocyclic compounds, including some substances that cannot be produced by conventional methods. In view of this fact, the authors of the study reported herein worked to develop a preparative method of producing it. They found that by reacting 2,2'-dioxadiethyleneoxadiphenol with phosphorus trichloride in the presence of tert-butyl alcohol under conditions of high dilution it is possible to obtain the title crown ether in a 50% yield. Studies of its chemical properties led the authors to conclude that its reactivity is very different from that of its closest analogue, diphenylphosphite. It undergoes Arbuzov's reaction with chlorane to form 2,2,2-trichloromethyl-1-oxyethylphosphonate. It undergoes a Todd-Atherton reaction with amines as easily as diphenylphosphite does. In all, the authors synthesized 10 compounds and subjected them to elemental analysis and IR, <sup>1</sup>H and <sup>31</sup>P NMR spectroscopy. The data obtained from these studies are summarized in a table. Also included are details of the procedures followed in synthesizing five of the compounds discussed. Table 1; references 8: 6 Russian, 2 Western.

#### The Reaction of Acid Chlorides of Phosphoric Thioacids With Imino Ester Hydrochloride of Isobutyric Acid. Properties of Phosphorylthioimidates

917M0162E Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript  
received 26 Apr 89) pp 384-387

[Article by Yu.I. Krongauz, V.V. Potapova, V.B. Sandakov, T.N. Koroleva, and L.M. Bogomolova, All-Union Scientific Research Institute of Chemical Plant Protectants, Moscow]

UDC 547.241

[Abstract] The authors of the study reported herein decided to examine the reaction of acid chlorides of phosphoric thioacids with hydrochlorides of imino esters of carbonic acids. They did so by way of the example of the reaction of hydrochloride of iminoethyl ester of

isobutyric acid and O,O-diethyl- and O-ethyl-O-4-chlorophenylchlorothiophosphates. The reaction was conducted in chloroform and dioxane at 40 to 50°C for 4 to 5 hours with a reagent ratio of 1:1 to 1.3:1.5 to 2.2. The experiment results led the authors to hypothesize that the formation of amidothiophosphates and ethyl ester of isobutyric acid is the result of the hydrolytic instability of the phosphorylimidates, i.e., hydrolysis at the C=N bond. The presence of isobutyramide in the reaction mass was explained by (1) the decomposition of hydrochloride of iminoethyl ester of isobutyric acid under the reaction conditions, which is in complete agreement with published data, and by (2) the hydrolysis of the phosphorylimidates at the P-N bond. References 9 (Russian).

#### Synthesis and Properties of Phosphorylated 2,6-Di-Tert-Butyl-4-Methylene-2,5-Cyclohexadienones

917M0162F Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript  
received 28 Jul 89) pp 387-391

[Article by R.K. Ismagilov, V.V. Moskva, V.P. Arkhipov, A.Ye. Ivantsov, and L.Yu. Kopylova, Kazan Chemical Technology Institute imeni S.M. Kirov]

UDC 547.564+547.26'118

[Abstract] The authors of the study reported herein examined the oxidation of phosphorylated spatially inhibited phenols synthesized on the basis of an Arbuzov reaction by reacting 2,6-di-tert-butyl-4-dimethylaminomethylphenol or 3,5-di-tert-butyl-4-hydroxybenzyltrimethylammoniumiodide with esters of P (III) acids at temperatures between 140 and 190°C. The said reaction was found to result in the intermediate formation of phenoxy radicals and to eventually result in the formation of 4-methylene-2,5-cyclohexadienones. The reaction of amines (alinine and piperidine) with phosphorylated 4-methylene-2,5-cyclohexadienones was found to result in 1,6-adducts. The compounds synthesized were subjected to the following studies: IR spectroscopy (using a Specord M-80 spectrometer), <sup>1</sup>H NMR spectroscopy (using a Tesla BS-567A spectrometer with a frequency of 100 MHz), <sup>31</sup>P NMR spectroscopy (using a Bruker WP-80 spectrometer), and EPR spectroscopy (using an RE-1306 spectrometer). The procedures followed in synthesizing all of the compounds studied are detailed along with yields and spectroscopic study results. Figures 2; references 5: 3 Russian, 2 Western.

#### The Reaction of Arylaliphatic 1,4-Diketones With bis(Trimethylsilyl)hypophosphite

917M0162G Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript  
received 17 Jul 89) pp 391-396

[Article by V.I. Vysotskiy, S.V. Biryukov, M.F. Rostovskaya, B.K. Vasilyev, S.V. Lindeman, and Yu.T. Struchkov, Far Eastern State University, Vladivostok]

UDC 547.438.1+547.442.4

[Abstract] The authors of the study reported herein reacted 1,4-diketones with bis(trimethylsilyl)hypophosphite and subjected the resultant compounds to elemental analysis and a series of spectroscopy (IR, PMR, NMR) studies. They discovered that even in the absence of catalysts, 1,4-diketones begin to react with the reagent bis(trimethylsilyl)hypophosphite at ambient temperature. Two types of products form after the reaction mixtures break down: linear adducts of two hypophosphite molecules and one molecule of diketone and 2,5-dihydroxyphospholane. The hydroxyl groups occupy the trans position in the phospholane ring. The reaction products were isolated either directly or else in the form of methyl esters obtained by processing the hydrolyzed reaction mixtures with diazomethane. Mixtures of stereoisomer methyl esters formed when the individual acids were subjected to methylation with diazomethane. Melting points and elemental analysis results are summarized in tabular form for selected compounds synthesized:  $C_{16}H_{20}O_6P_2$ ,  $C_{22}H_{24}O_6P_2$ ,  $C_{22}H_{21}O_4P$ ,  $C_{18}H_{24}O_6P_2$ ,  $C_{17}H_{19}O_4P$ ,  $C_{24}H_{28}O_6P_2$ ,  $C_{23}H_{23}O_4P$ , and  $C_{26}H_{31}O_4PSi$ . Tables 2; references 5 (Russian).

#### Phosphorylation of 5-Alkyl-4,5-Dihydro-1,2,4-Triazine-6-Ones

917M0162H Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript received 19 Jan 90) pp 406-415

[Article by Zh. Sapozhnikova, A.F. Prokoveva, V.N. Volkova, V.V. Negrebetskiy, L.A. Pokrovskaya, and N.N. Melnikov, All-Union Scientific Research Institute of Chemical Plant Protectants, Moscow]

UDC 547.241

[Abstract] In an attempt to find additional biologically active substances, the authors of the study reported herein attempted to synthesize a series of phosphorus-containing derivatives in the series 5-alkyl-4,5-dihydro-1,2,4-triazine-6-ones. They phosphorylated a series of phosphorus-containing derivatives in the series 5-alkyl-4,5-dihydro-1,2,4-triazine-6-one with acid chlorides of phosphoric and thiophosphoric acids under soft conditions in acetonitrile at temperatures ranging from 35 to 50°C in the presence of triethylamine or  $K_2C_3$  with the reagents in an equimolar ratio. The 5-alkyl-4,5-dihydro-1,2,4-triazine-6-ones were phosphorylated by the phosphoric and thiophosphoric acid chlorides at the  $N^4$  atom and, in some cases, at the O and  $N^4$  of the triazine ring. The formation of bis-phosphorylated triazines was explained by the presence of a tautomeric oxy-form in the starting triazines under the given reaction conditions. Data from the IR and ( $^1H$ ,  $^{13}C$ , and  $^{15}N$ ) NMR spectroscopic studies to which the synthesized derivatives were subjected are presented in tabular form and discussed in detail. Tables 3; references 6: 3 Russian, 3 Western.

#### Synthesis of Diphosphine Monoxides

917M0162I Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript received 23 Jul 90) pp 506-507

[Article by N.N. Demik, M.M. Kabachnik, Z.S. Novikova, and I.P. Beletskaya, Moscow State University imeni M.V. Lomonosov]

UDC 546.18+547.241

[Abstract] The authors of this brief letter to the editor report that they have demonstrated that dialkylchlorophosphines react with complete ethers and etheramide acids of trivalent phosphorus in the presence of catalytic quantities of Lewis acids ( $NiCl_2$  and  $SnCl_2$ ) at 150°C over the course of 30 minutes. They succeeded in obtaining monoxides of asymmetric diphosphines with a yield of 96 to 98%. Data on five of the compounds considered in their research are included. References 2: 1 Russian, 1 Western.

#### Photoelectron Spectra of C,C-bis(Dialkylamino)methylenephosphines

917M0162J Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript received 22 Jun 90) pp 509-510

[Article by V.I. Vovna, L.N. Alekseyko, B.V. Okhota, A.V. Ruban, V.D. Romanenko, and L.N. Markovskiy, Scientific-Research Physical Technical Institute, Far Eastern State University, Vladivostok, and Organic Chemistry Institute, UkSSR Academy of Sciences, Kiev]

UDC 539.194:546.18

[Abstract] The authors of this brief letter to the editor used the technique of photoelectron spectroscopy to study the n- $\pi$  reaction that is important in explaining the chemical properties of phosphalkene  $(Me_2N)_2C=NH$  and three phosphalkenes with the general formula  $R_2N(R'_2N)C=PH$  (where  $R = R' = Me$  (II), Et (IV);  $R = Me$ ,  $R' = Et$  (III)). The four highest filled molecular orbitals of the first compound (the  $\pi_{CN}$  and the  $n_N$  orbitals) were found to correspond to the bands with the photoelectron spectra having maxima at 8.48, 9.74, and 11.47 eV. The first was related to the two  $n_N$ -orbitals of the amino fragments, the second to the  $n_N$ -orbital of the imino nitrogen, and the third to the  $\pi_{CN}$ -orbital. The absence of any marked splitting of the two n-levels of the amino groups as opposed to what occurs in the compound  $(Me_2N)_2C = CH_2$  confirmed the interaction of both combinations of n-orbitals of the amino groups with the  $\pi$ - and/or n-orbital of the amine fragment. The mixing of the three n-orbitals was also shown to increase as the n- $\pi$  conjugation in the acoplanar structure decreases. In the spectrum of methylenephosphine, the three nonbinding electron pairs and  $\pi$ -electron were found to correspond to the first four bands with maxima at 7.26, 8.26, 9.44, and 10.58 eV. These bands were

analyzed on the basis of their oscillation contours, relative intensity, and photoelectron data. Replacement of the methyl groups by ethyl groups resulted in the destabilization of all four molecular orbitals. This change is analyzed along with changes in the intramolecular  $n_N-n_P$  and  $n_P-\pi_{CP}$  interactions in the three phosphalkenes with the general formula  $R_2N(R'_2N)C=PH$ . References 3: 1 Russian, 2 Western.

#### Stable Alkoxyfluorophosphoranes

917M0162K Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript  
received 16 Apr 90) pp 510-511

[Article by O.I. Kolodyazhnyy and S.N. Ustenko, Institute of Bioorganic Chemistry and Petrochemistry, UkSSR Academy of Sciences, Kiev]

UDC 547.241

[Abstract] The authors of this brief letter to the editor report on their discovery that phosphorus fluoroilides attach alcohols at the P=C bond, which in turn results in the formation of stable alkoxydialkyldifluorophosphoranes that, unlike most known compounds of this type, are completely stable and may be isolated individually. The stability of the resultant alkoxydialkyldifluorophosphoranes was attributed to the existence of spatial impediments in the molecule that prevent the alkoxyl group from proceeding into an axial position and breaking the O-C bond. Only upon heating to temperatures above 100°C did the alkoxydialkyldifluorophosphoranes form fluorophosphinates and alkylfluorides with a

quantitative yield. The reaction of the phosphorus fluoroilides with alcohols was thus determined to be a convenient method of replacing a hydroxyl group with fluorine atoms. References 2 (Russian).

#### The Reaction of Sodium Dithionite With Organyltrichlorophosphoniumhexachlorophosphorates

917M0162L Leningrad ZHURNAL OBSHCHEY  
KHIMII in Russian Vol 61 No 2, Feb 91 (manuscript  
received ) pp 513-514

[Article by V.V. Kormachev, Yu.N. Mitrasov, and Ye.A. Anisimova

UDC 547.341

[Abstract] Others have demonstrated the possibility of using sodium thiosulfate and pyrosulfite to produce diacid chloride of styrylphosphonic acid from an adduct of phosphorus pentachloride with alcohol. The elevated temperature of the process (70°C), its duration, and its ambiguity in the case of sodium thiosulfate all reduce the preparative capabilities of this reagent, however. The authors of this brief letter to the editor report that according to their research, sodium dithionite is devoid of these shortcomings. They discovered that reacting sodium dithionite with organyltrichlorophosphoniumhexachlorophosphorates in a medium of inert organic solvent at room temperature results in good outputs of diacid chlorides of organylphosphonic acids. The reaction proved to be weakly exothermal, with a temperature of 5 to 10°C being observed. The process was found to be completed within 10 to 30 minutes after the sodium dithionite was added. The structure of the diacid chlorides was confirmed by IR and  $^1H$  and  $^{31}P$  NMR spectroscopy, and their constants were found to be in good agreement with those in the literature. References 3 (Russian).

**New Multifunctional Lube Oil Additive**

917M0153A Moscow *KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 4, Apr 91 pp 5-6*

[Article by S. B. Borshchevskiy, Ye, V. Shabanova, A. A. Fufayev, F. N. Merzlikin, and O. V. Belyayeva, Petroleum Refining SRI (A-U), Moscow]

UDC665.763-404.038.5

[Abstract] Dithiophosphates of metals, especially zinc, are widely used as additives to improve lube oils. In the USSR these additives are produced in two forms, DF-11 and VNII NP-354, the former being the most prevalent. DF-11 (zinc dialkydithiophosphate) is included in the composition of motor oils Groups V, G, and D. It is also used in industrial oils and hydraulic fluids. The volume of consumption of these additives keeps growing each year both in the USSR and abroad. However, more severe service conditions and the extensive use of low viscosity oils elevated the quality requirements of various additives, including zinc dithiophosphates. Thus a new additive, boron-modified dialkydithiophosphate, DFB (TU 38.1011131) was developed. The present work lists its physical properties, composition, results of anti-corrosion testing, and resistance to pitting and wear. While retaining the properties of zinc dithiophosphate, the new additive exceeds it in terms of friction and pitting effectiveness. Figures 2; references 5 (Russian).

**Antiknock Quality of Alcohol Based Synthetic Motor Fuels**

917M0153B Moscow *KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 4, Apr 91 pp 8-10*

[Article by Yu. M. Gulyamov, V. A. Gladkikh, Yu. V. Shtefan, and V. D. Malykhin, Chemical Technological Institute, Dnepropetrovsk; State Chemistry SRI]

UDC621.434:662.754

[Abstract] The lower alcohols appear promising both as an alternate motor fuel and as a means to decrease the toxicity of exhaust gases. The chief advantage of alcohols lies in the possibility of synthesizing them from any carbon-containing source, such as natural gas, coal, shale, and agricultural and industrial wastes. Alcohols also have high antiknock capability, making it possible to raise the compression ratio of an internal combustion engine, thereby making it more efficient. Using alcoholic fuel, an engine may operate using a lean fuel mix with an excess of air, thereby increasing its economy and emitting less toxic exhaust gases. As an alternative to petroleum fuels, alcohol mixtures used as antiknock additives does not solve the main problem, i.e. substitution of petroleum based motor fuels. It is thus more rational to use mixtures of alcohols as a fuel base. The drawbacks of such a fuel include poor starting and low energy which may be overcome by using dimethyl and diethyl ethers and low boiling point petroleum fractions as additives.

Among the energy additives interest now centers on synthetic hydrocarbons and low octane straight run gasoline fractions and fractions resulting from thermal cracking of bottoms stocks. Development of this type of fuel would enable broader utilization of the primary products of petroleum refining, decrease the high aromatic component fraction in fuel blends, and make possible the rejection of toxic tetraethyl lead. A promising source for the development of an alcohol base and a synthetic hydrocarbon additive could be metallurgical industry off-gases, now being burned off at the flue. This could provide 20-25 percent of the annual motor fuel requirements in high octane fuel. In the present work a study was made of the antiknock qualities of gasohol mixtures containing (percent by weight) C<sub>1</sub> 50-70; C<sub>2-4</sub> 20-40; C<sub>5</sub> and higher 5-8; water 2-3. Hydrocarbon components included straight run gasoline fractions and a synthetic hydrocarbon mix obtained by bi-functional catalysis of synthesis gas. Octane number (both motor and research methods) for the blends are listed. Figures 3; references 7 (Russian).

**Production of Non-Ethyl Gasoline AI-93 at Type 1-B Unit**

917M0153C Moscow *KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 4, Apr 91 pp 13-14*

[Article by M. I. Rustamov, A. D. Guseynova, A. M. Guseynov, S. M. Asker-Zade, A. B. Kuliyeu, Y'e. I. Pryanikov, and A. A. Mkrttychev, Petrochemical Processing Institute imeni Yu. G. Mamedaliyev, Baku; Novobakinsk Petroleum Refinery imeni Vladimir Ilich]

UDC665.644.2

[Abstract] Following a design developed at Azgiproneftekhim, a Type 1-B catalytic cracking unit at the Novo-Bakinsk Oil Refinery was rebuilt to produce non-ethyl gasoline AI-93. The catalytic cracking reactor now consists of a transfer duct (20 m length, 0.6 m diameter) terminating in a cylindrical vessel 1.6 m in diameter and 5.5 m high. The catalyst regenerator is a 28 m vertical duct of varying cross section. Inside the regenerator are two grid plates. The transfer line of the regenerator is separated above the fluidized bed to facilitate countercurrent regeneration at 80-100 °C, which in turn precludes spontaneous combustion of CO to CO<sub>2</sub>. In addition to rebuilding the reactor-regenerator unit, a gas fractionating unit was assembled for stabilizing the AI-93 gasoline. After rebuilding, the unit was operated under various refining conditions. The results of its operation in refining non-ethyl gasoline AI-93 from a vacuum distillate are discussed in the present work. Figures 3; references 3 (Russian).

**Change in Colloidal Stability of Functioning Motor Oils Contaminated with Coolant**

917M0153D Moscow *KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 4, Apr 91 pp 19-22*

[Article by V. A. Artemyev, D. V. Boykov, and V. L. Lebedeva, Yaroslav Motor Plant Production Association "Avtodizel"]

UDC621.892.096-714:547.91

[Abstract] Colloidal stability of motor oils depends largely on the properties of the base oil and the composition of any additives and contaminants, especially from coolants. In many cases, oil contamination with coolant liquids (water and antifreeze) results in lower operational dependability of internal combustion engines owing to higher sludge and carbon deposits, and a drop in the capacity of the oil filter. This is especially true for oils having unsatisfactory hydrolytic stability during storage, e.g. oils M-10V<sub>2</sub> and M-10G<sub>2</sub>, which contain additives subject to hydrolysis. Operational experience with YaMZ diesel engines using M-10G<sub>2</sub>(k) and M-10D(m) lube oils revealed cases of lowered colloidal stability and loss of overall stability of additive composition on entry of water into the crankcase. In the present work a study was made of the changes in operating properties of motor oils during bench testing of engines with coolant liquid added artificially to the lubricating system. Also, a laboratory method was developed for determining the threshold amount of water in lube oil without causing sedimentation of contaminants and additives. Operational experience with the YaMZ engine shows that if the cooling system is air-tight, the water content in the oil does not exceed 0.03 percent and consists basically of condensed water from the combustion products. The results also underscore the necessity of raising the colloidal stability of motor oils to the level of foreign analogs. Figures 3; references 12: 10 Russian, 2 Western.

#### Selection of Conditions for Optimum Action of Metal-Containing Additives in Lube Oils

917M0153E Moscow *KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 4, Apr 91 pp 24-25*

[Article by I. N. Yevdokimov and N. Yu. Yeliseyev, Petrochemical and Gas Industry Institute imeni I. M. Gubkin, Moscow]

UDC665.733.5

[Abstract] The anti-friction properties of lubricating oils may be improved by using additives containing organic substances and metallic salts, for example copper sulfate dissolved in glycerine, or additives containing salts of tin, silver, gold, or other metals dissolved in polyhydric alcohols or ethers. Employing these additives cuts the coefficient of friction 10-16 fold and wear 8-10 fold. However, despite the demonstrated effectiveness of the metal-containing additives, they are not yet widely used owing to the dearth of information on the optimum conditions for displaying anti-friction properties. This requires knowing the best ratio of additive component concentrations and the optimal concentration of additive in the lube oil. Optimization of tribological processes includes a large number of interacting parameters. To solve this problem, use of systems analysis methods by subdivision into sub-problems has been recommended. In respect to complex liquid lubricants, i.e.

those containing additives, suitable sub-problems could comprise: 1) identification of physical chemical processes taking place during preparation; 2) study various lubricant compositions which provide minimum resistance in standard friction pairs; 3) study the evolution of system parameters during operation of real friction nodes, e.g. oxidation and heat resistance of liquid lubricants, wear, and corrosion of friction pairs. Problem 1) was worked out using a tin additive in a previous work where it was established that surface-active molecular complexes are formed with the additive during the preparation stage. In the present work some results of problem 2) using the same additive are presented. The anti-friction properties of the additive in lube oil I-40A during operation of a steel-steel friction pair was studied. Figures 3; references 6: 5 Russian, 1 Western.

#### Evaluating Economic Effectiveness of Steps Taken To Prevent Air Pollution at Oil Refineries and Petrochemical Enterprises

917M0153F Moscow *KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 4, Apr 91 pp 27-29*

[Article by A. M. Zeninskiy, VNIIS]

UDC338.2:628.854.3

[Abstract] Standard methods for evaluating economic loss due to environmental pollution and the economic effectiveness of steps taken to safeguard the environment fail to take into account the new operating conditions of oil refineries and petrochemical enterprises as well as payments made for contaminating the atmosphere. A proposed method for computing the effectiveness of expenditures for safeguarding the atmosphere was developed to take into account both the fundamental positions of the method as well as industrial recommendations and instructions. The need for air pollution control steps is established at the pre-production stage and its optimum variant is selected while shaping research and experimental-design operations, and it is at this stage that any expected economic effect is determined. During the production stage, the effects of atmospheric control measures on the cost-accounting indicators of the enterprise are evaluated by taking into account the actual (planned) economic effect. The economic effect  $E_t$  of atmospheric control measures over the period of time  $t$  is characterized by comparing the economic results  $R_t$  with the financial outlay  $Z_t$  required to carry out these measures:  $E_t = R_t - Z_t$ . Other formulas are presented for determining annual loss in profits and the effects of capital investment. References 10 (Russian).

#### Metal-Resistant Cracking Catalysts

917M0153G Moscow *KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 4, Apr 91 pp 31-32*

[Article by T. S. Kostromina and R. R. Aliyev, Petroleum Refining SRI (A-U), Moscow]

UDC665.644.2:661.183.6

[Abstract] With the increasing use of heavy crudes and even bottom stocks for catalytic cracking, the problems of heavy metal catalyst poisoning become more and more critical. Nickel, vanadium, copper, iron and other metals present in the heavier crude fractions tend to lower the activity and process selectivity of the catalyst, resulting in lower yields of gasoline and greater amounts of light gases and coke. Zeolite-containing catalysts are known to be resistant to the poisoning effects of these metals, although they also lower cracking selectivity and increase catalyst attrition. The matrix plays an important role in metal-resistant catalysts. A matrix having correct physical and mechanical properties is capable of adsorbing part of the metals present in the crude, thereby precluding their entry into the zeolite and thus destroying it. Domestic cracking catalysts employ silica-alumina hydrogel which has a highly developed fine pore structure. Substituting the silica-alumina hydrogel with open-pored kaolin clay raises the catalyst's resistance to metal poisoning. In the present work a study was made of the effects of changes in the quality and quantity of zeolite on the activity of a catalyst before and after metal poisoning. A series of catalysts were prepared from GMTs catalyst (clayey metal-resistant zeolite) containing 5-30 percent by weight zeolite. Nickel and vanadium were impregnated onto the catalysts in 1 to 2 ratio. The results confirm the suitability of GMTs as a metal-resistant catalyst. Figures 3; references 3: 2 Russian, 1 Western.

#### VK-Zeolites - Promising Sorbents for Sulfur Refining of Petroleum Gases

917M0153H Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 4, Apr 91 pp 32-33

[Article by Z. K. Olenina, N. P. Moreva, Yu. P. Yasyan, and A. Yu. Adzhiyev, Gas and Oil Design SRI (A-U)]

UDC541.183.03

[Abstract] The problem of refining petroleum gases containing mercaptans in addition to hydrogen sulfide arose with the opening up of the Tengiz, Zhanazhol, and Malgobek-Voznesenskiy oil fields. Natural gases are de-mercaptanized with NaX zeolite, which has a good sorption capacity for mercaptans. However, it also has a high enough dynamic capacity for C<sub>3</sub> and higher and hydrocarbons which precludes its use for refining petroleum gases. Co-adsorption of the heavier hydrocarbons results not only in their loss, but also in a lower mercaptan capacity. Moreover, NaX, having a silicate modulus of about 3, breaks down in acid media resulting in poor adsorption properties and low service life. The effectiveness of adsorption refining of gases depends largely on the sorbent and its resistance to heat and acids. Synthetic high silica zeolites (VK-zeolites) used as catalysts in petrochemical processes have a silicate modulus ranging from 12 to 1000 and therefore have high heat and acid resistance as well as being hydrophobic. In

the present work a study was made of the adsorption capacity of high silica zeolite catalysts TsVK and TsVM, domestic analogs of zeolite ZSM, to determine the feasibility of using them to de-mercaptanize petroleum gas. Over 20 catalyst samples were tested. Compared to NaX, these catalysts have a higher dynamic capacity for ethyl mercaptan and they are "inert" to heavy hydrocarbons. This enables more complete utilization of sorbent capacity for the target components while at the same time lowering the sorbent charge. It may also be assumed that they will have a longer service life, owing to high heat, acid and mechanical resistance. Figure 1; references 2 (Russian).

#### Electrolytic Separation of Hydrocyanic Acid From Coke Oven Gas Final Cooling Cycle Water

917M0164A Moscow KOKS I KHIMIYA in Russian No 6, Jun 91 pp 23-26

[Article by V. D. Barskiy, T. N. Gnezdilova, O. V. Fedulov, V. A. Shlifer, V. N. Rubchevskiy, Yu. A. Chernyshov, and S. N. Danilov, Zaporozhskiy Coke-Chemical Plant]

UDC662.764.074

[Abstract] Closing the final cooling cycle in coke oven gas production is one possible means of preventing toxic gas venting to the atmosphere. Although this completely excludes harmful wastes from the atmosphere, it also worsens operation of benzene separation stage in the de-sulfurizing section resulting in a rise in the contents of hydrogen cyanide, ammonia, hydrogen sulfide, and other gases in the coke oven gas. Therefore, studies directed toward cleansing the final cooling cycle water, especially from hydrogen cyanide, are urgent. Most effective are methods of oxidative decomposition with hypochlorous acid and subsequent flocculation with addition of iron salts. However, these methods run into difficulties when applied to recycled water owing to insufficient reaction selectivity of the reagents. In the present work a feasibility study was made of a process wherein the cyanogen is electrolytically bound up with iron ions from a soluble anode followed by electrocoagulation of the resulting insoluble complexes, and electro-flotation of the latter. The first stage of the study was done on a batch basis in a rectangular box-type electrolyzer employing a Ct-3 anode and a stainless steel cathode, and fitted with a connecting pipe to remove accumulated pulp. This provided information on water parameters (cyanogen concentration, pH, and temperature), design features (electrode material and distance between), and process operating characteristics (water treatment and standing times, current density, electrolyte and surfactant addition). Questions relating to utilization of the solid phase will be dealt with in a future report. Figures 4; references 5: 4 Russian, 1 Western.

### Technology for Preparing Gas-Cleansing Catalysts From Metallurgical Slag

917M0164B Moscow KOKS I KHIMIYA in Russian  
No 6, Jun 91 pp 28-30

[Article by L. B. Pavlovich, Ye. A. Zolotukhin, Ye. I. Andreykov, and A. P. Romanenko, Eastern Coal-Chemical SRI]

UDC662.74.013:628.53

[Abstract] One method for stripping gases of pollutants is thermal or catalytic deep oxidation to CO<sub>2</sub> and water at 1100-1200°C. Oxides of nitrogen are formed and the level of benzpyrene stripping remains low unless the temperature is raised to 1500-1700°C, requiring considerable energy outlay. Catalytic oxidation is effective, although it is encumbered with catalyst poisoning by H<sub>2</sub>S, SO<sub>2</sub>, carbon, and coal dust. Stationary bed catalytic stripping may be used in the condensation, sludge removal, coal tar, coke cake, and other units. However, the catalysts employed in these units in the USSR are costly and in short supply. Bulk catalysts are developed on a  $\gamma$ -alumina base, which is also in short supply. A catalyst system was developed at the Kuznetsk Branch of the Eastern Coal-Chemical SRI which is based on metallurgical slag containing oxides of silicon, aluminum, calcium, and transition metals in addition to the base metals. These catalysts are low in activity owing to their small specific surface and the presence of the transition metals, although they may be modified to increase catalytic activity for deep oxidation. In the present work a pilot plant process is described for producing these catalysts which are said to cost 2,000 to 5,000 rubles per ton, depending on grade, while industrial catalysts cost 3,600 to 60,000 rubles per ton. Figures 2; references 14 (Russian).

### Venturi Scrubbers - Used to Cool and Cleanse Coke Oven Gas

917M0164C Moscow KOKS I KHIMIYA in Russian  
No 6, Jun 91 pp 30-31

[Article by V. G. Vshivtsev, N. B. Volgina, and A. N. Starodubtsev, Eastern Coal-Chemical SRI, Sverdlovsk]

UDC662.764.045.5

[Abstract] The operational stability of the heating and product recovery systems in coke oven batteries is a function of the efficiency and dependability of gas cleansing units. Using low pressure Venturi scrubbers provides the most economical means for the achieving required degree of gas purity. These units provide for coagulation and settling of aerosols and other volatile coke oven gas components as well as saturation with water vapors. Capital investment is 4-8 times less than electric filtration. Other advantages include combining the cooling and gas stripping processes, lack of explosion hazard, simple assembly and maintenance, option of

on-site assembly, compactness, and low metal volume. High dependability is provided by a special system for dispersing recycled water and an appropriate selection of pumps and heat exchangers. The ecological cleanliness, possibility of automation, high unit capacity, broad range of operational stability, and absence of deposits in the equipment at any gas temperature or composition, make these Venturi units attractive for the coke-chemical industry. The Eastern Coal-Chemical SRI developed and installed units for cooling and cleansing coke oven gas of suspended particles of resins and naphthalene in low pressure Venturi scrubber jets, which provide 80-90 percent purity of coke oven gas; second stage gas cooling and wet filters provide 92-98 percent purity; absorbers remove naphthalene, ammonia, and hydrogen sulfide. Capital expenditure for fitting out a 100,000 cubic meter per hour unit is below 80,000 rubles. Venturi scrubbers have been installed at Kaliningrad, Gubakha, Novolipetsk, and Kemerov coke oven plants. Currently, the Eastern Coal-Chemical SRI is independently designing and developing custom-made units for clients. Figures 4.

### Ozonization of Effluents Following Two-Stage Biochemical Treatment

917M0164D Moscow KOKS I KHIMIYA in Russian  
No 6, Jun 91 p 42

[Article by D. R. Kazakevich, I. B. Yevzelman, and Ye. A. Ptitsyna; Far Eastern Coal-Chemical SRI, Sverdlovsk Branch Railroad]

UDC662.74:662.793.5

[Abstract] Owing to the widespread use of dry quenching in the coke-chemical industry, a problem arose in the utilization of biochemically treated industrial effluents, previously used in wet quenching. These effluents are best recycled. However, the presence of up to 600 mg per liter of suspended matter in biochemically treated waste could result in biological growths in the cooling towers and heat exchangers. These growths may be prevented through chlorination or ozonization. The former is less costly, although it produces toxic and cancerogenic compounds which are highly resistant to biochemical oxidation and highly corrosive. Ozone, a powerful and almost instantaneous oxidant, facilitates biochemical pretreatment of the effluent, rendering it lighter in color and turbidity, and eliminating unpleasant odors. A study was made using a coke-chemical effluent containing (in mg per liter) 2.5 phenols, 3.8 thiocyanates, 1250 chemical oxygen equivalent, and pH 8.3. This was treated with 6-200 mg ozone per liter. The results indicated that ozone is effective at doses starting at 50 mg per liter with no further increase in activity at higher dosages. The possibility of secondary growth of biomass was confirmed. Analysis showed that *Pseudomonas* bacteria is the most resistant to ozone. Thus, ozonization must not

be considered as being fully effective in preventing growth of biomass in coke-chemical effluents when used as recycled water.

**New Methods for Treating Coke Oven Gas  
(Proceedings of Sector Seminar)**

*917M0164E Moscow KOKS I KHIMIYA in Russian  
No 6, Jun 91 pp 47-49*

[Article by T. G. Anikina and S. M. Listova, Eastern  
Coke-Chemical SRI]

UDC662.74.001.7(047)

[Abstract] The title seminar, held in November 1990 at the Eastern Coke-Chemical SRI, was attended by representatives of 10 coke-chemical enterprises and 33 research, design, and academic institutes. The basic task of the seminar was to gather together the most outstanding work experiences and new technological findings, analyze the ecological status of coke-chemical production, and consider the prospects for incorporating new technological processes for treating the gases. The

participants observed that currently there exists a considerable volume of know-how which should be employed in the rebuilding and building of new technological processes for treating coke oven gas. References 16 (Russian).

**Use of Brown Coal Coke for Treating Stack Gases  
and Minimization of Incinerator Residues**

*917M0164F Moscow KOKS I KHIMIYA in Russian  
No 6, Jun 91 pp 50-51*

[Article by D. A. Tsikarev, Mined Fuels Institute]

UDC665.7.032.54(047)

[Abstract] Information has been assembled in West Germany on the burning of wastes from automated combustion units and high productivity units for treating stack gases. Further increases in the effectiveness of these methods for safeguarding the environment lie in the use of adsorption filters and thermocatalytic methods of treating wastes. One of the most effective and least costly adsorbent filter materials is coke obtained from Rheine brown coals in annular furnaces. Physical and chemical properties of the coke are presented.

**Dissolution Process of Butadiene Nitrile Carboxylated Latex Particles as Determined by Dynamic Raman Spectra**

917M0155A Moscow KAUCHUK I REZINA in Russian No 4, Apr 91 pp 9-12

[Article by S. A. Blokhtina, A. S. Bukin, and V. V. Klyubin]

UDC541.182.65.046

[Abstract] Specific properties of a latex copolymer are largely determined by the carboxyl groups present. The dispersed composition of these latexes depend greatly on the hydroxyl ion concentration in the dispersion medium. In acid media, these latexes are in the form of dispersions of dense globules. As the pH is increased, the carboxyl groups become neutralized and negative charges appear on the macromolecular chain causing partial hydrophilization of the latex polymer, and swelling of the globules, thereby making it possible to use these lyophilic colloids as thickeners, dispersing agents, flocculants, and emulsifiers. While the literature contains many citations on the properties of carboxyl-containing copolymer latexes obtained by electron microscopy, viscosimetry, and turbidimetry, only a few refer to dynamic Raman spectra, although the latter is the most convenient method. In the present work this method was used to study the structural changes in the dispersed phase of a latex having a high content of carboxyl groups (latex BNK-20/35) as the hydrogen ion concentration in the dispersion medium was altered. Quantitative data were obtained on the changes in the dispersed composition of the carboxylated latex which supplement and refine the qualitative concept of the dissolution process derived from other techniques. The results may serve as a link between the method of preparing a latex and its technological properties. Figures 4; references 8: 6 Russian, 2 Western.

**Effect of Nature of Filler on Properties of Low Module Cellular Rubbers**

917M0155B Moscow KAUCHUK I REZINA in Russian No 4, Apr 91 pp 12-13

[Article by N. V. Savelyeva, V. Ye. Denisenko, and N. A. Sabadyr]

UDC678.4:678.763

[Abstract] Currently popular aquatic sports requires wet suits made of cellular rubber reinforced with an elastic liner. The serially produced cellular rubber used for this purpose contains a technical grade carbon P803 as filler. However, introduction of a filler affects the rubber's relaxation properties owing to stress relaxation at the polymer-filler interface causing a softening effect in both the monolithic and cellular rubbers. Evidently, physical processes taking place in low-modular rubber determine

the wearability of the wet suit under severe wear conditions. Therefore, a study was made of the effects of the nature of the filler on the relaxation properties of a low-modular (chloroprene) cellular rubber mercaptan grade DN. Fillers used were lithopone, chalk, kaolin, silica BS-50, and technical carbon P803; other ingredients remained constant. Test results demonstrated that cellular rubber containing lithopone is the most low-modular and elastic, although it has less tensile strength than the others. The results further indicated that lithopone (for light-colored rubbers) or carbon P803 (for dark rubbers) should be included in the formulas for cellular rubbers requiring rapid recovery after a given dynamic elongation. Figures 3; references 2 (Russian),

**Electrically Conducting Rubbers Based on Polymerization-Filled Silicon-Organic Elastomers**

917M0155C Moscow KAUCHUK I REZINA in Russian No 4, Apr 91 pp 13-15

[Article by Dzh. N. Aneli, D. G. Pagava, Ts. V. Kakuliya, and N. I. Tsomaya]

UDC537.31.678:063.678.01

[Abstract] Rubber compositions prepared by polymerization filling have improved properties as compared to conventional rubbers. Their microstructure is characterized with a more uniform distribution of filler particles in the polymer matrix. This method was used to prepare electrically conducting rubbers based on certain silicon-organic monomers and technical grade carbon P267-E. These rubbers also have several advantages over similar materials obtained by radiation or peroxide vulcanization. To further improve the electrical conducting properties of polymerization filled rubbers, a thermally split graphite (TRG) was utilized in the present work. The TRG was first treated with metallic potassium under argon to which was added octamethylcyclotetrasiloxane and heptamethylvinylcyclotetrasiloxane. After stirring vigorously, the mix was cooled, treated with water, and rolled into sheets. Conductivity and other tests demonstrated that polymerization filled compositions based on silicon-organic elastomers containing thermally split graphite have many useful service properties. The relatively low degree of filling and the high electrical conductivity enhances the elasticity of this material which may be used as a strain-sensitive component in devices subjected to sign-changing deformation. Figures 3; references 4 (Russian),

**Estimating Service Life of Elastomeric Components by Self-Heating Rate**

917M0155D Moscow KAUCHUK I REZINA in Russian No 4, Apr 91 pp 24-27

[Article by V. D. Savelyev, S. O. Lazarev, Yu. K. Mikhaylov, and V. A. Petrov]

UDC539.4.014

[Abstract] Elastic components of shock absorbers operate under cyclic conditions. In those cases where rubbers or other elastomers are used as the elastic component, it is possible for them to overheat and subsequently shorten their service life. While sufficient data now exist on the temperature status and the possibility of predicting the service life of such components, the needed experimental material is obtained by prolonged and costly testing. In the present work an alternative approach is considered that is based on the kinetics of the disruption. Formulas are presented which correlate dissipation energy with elastomer life time under cyclic load. A bond exists between the self-heating and disruption processes which is characterized by a change in the dissipative heating rate during service. An experimental method is proposed which is based on this bond between the parameters of dissipative heating and disruption. Figures 4; references 9 (Russian).

#### **Optimization of Rubber Compositions Based on Chlorosulfonated Polyethylene**

917M0155E Moscow KAUCHUK I REZINA in Russian No 4, Apr 91 pp 32-33

[Article by L. V. Abuzyarova, T. G. Samoylenko, and Ye. E. Potapov]

UDC678.4-678.066-678.6/8+677.041.65

[Abstract] Chlorosulfonated polyethylene rubber is a promising substance for making rubberized fabrics, since it is resistant to oil, gasoline, fire, ozone, weather, heat, frost, and wear. Rubberized fabric is complex composite material. One index of its quality is the strength of the rubber-to-fabric bond. A satisfactory level is normally achieved by preliminary saturation of the fabric with adhesives. However, this results in an increase in weight unacceptable for many applications, and a decrease in tensile strength on elongation. Modern polyamide technology also requires costly equipment and space. If type RU and ARU modifiers are used in diene rubber compositions, it is possible to raise the rubber-to-fabric bond strength in non-saturated polyamide and viscose fabrics. Furthermore, modifier RU also serves as vulcanizing agent for chlorosulfonated polyethylene. In the present work a study was made of the effect of filler BS-150, plasticizer KhP-1100, magnesium oxide vulcanizing agent, and modifier RU-1 on the adhesion, strength, and other properties of chlorosulfonated polyethylene rubber grade MR (TU 6-01-1-379-88). A specific formula is recommended based on the results. The rubber mix is equivalent in physical and mechanical properties to mixes based on Nairit used in rubberized fabric production. It exceeds them in respect to shear strength and frost resistance. References 3 (Russian).

### Radiation Destruction of Nitrocellulose

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[Article by A.T. Govorkov, Ye.A. Bannova, L.I. Volkhonskaya, G.Ye. Sokolova, and M.P. Fomina, Kemerovo State University]

UDC 541.14:547.458.82

[Abstract] The authors of the study reported herein conducted a series of experimental studies to determine the degree to which the radiation destruction of nitrocellulose is dictated by the nature of the starting cellulose, its degree of etherification, and the conditions of its irradiation. Nitrocellulose obtained from both cotton pulp and wood cellulose was used. The cellulose was nitrated by a mixture of sulfuric and nitric acids as described elsewhere. After nitration the nitrocellulose was stabilized by an ammonia solution and multiple water rinses until a neutral reaction was achieved. The nitrocellulose was then subjected to  $\gamma$ -radiation in an aqueous medium in air, in the form of a film in air, and in evacuated ampules. The irradiation was conducted on an MRX- $\gamma$ -20 unit at an absorbed dose rate of 1.39 Gy/s. The irradiation temperature was varied within the range from 35 to 85°. IR spectroscopy, capillary viscosimetry, and gas chromatography studies were performed along with chemical analysis and physicochemical tests. The radiochemical yields of the breaks of the bonds in the macrochain, the elimination of nitro groups, and the formation of gaseous radiolysis products were all determined. It was discovered that the nature of the starting cellulose has no effect on the destruction of nitrocellulose. The laws governing the radiation destruction of nitrocellulose under homogenous and inhomogeneous conditions were examined. The presence of a break in the curves plotted for the relationship  $(1/M_n - 1/M_{n0}) - D$  (where  $M_n$  and  $M_{n0}$  are the average molecular masses before and after irradiation and  $D$  is the absorbed dose in kGy) is critical in the radiochemical processes occurring in nitrocellulose. This break is the result of secondary processes that occur differently depending on irradiation conditions. When nitrocellulose is irradiated in an aqueous medium, for example, the nitrogen dioxide evolved during radiolysis dissolves and results in the formation of nitric and nitrous acids. The diluted acids do not, for practical purposes, affect the stability of the nitrocellulose. Only concentrated mineral acid solutions are destructive. The authors hypothesize that the appearance of a break in the aforementioned dependence is caused by both radiation and chemical destruction of the nitrocellulose due to hydrolysis. Increasing the temperature also accelerates the chemical destruction of nitrocellulose, thus increasing the radiochemical yield under given conditions. When nitrocellulose is irradiated in film form, the break in the aforesaid dependence appears at absorbed doses of 550-600 kGy, which corresponds to a deep degree of destruction. Figures 4, table 1; references 12: 11 Russian, 1 Western.

### The Effect of $\gamma$ -Irradiation of Aluminosilicate Hydrogels During the Process of Their Preparation on the Composition of Gasolines Produced by Cracking Light Gas Oil

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[Article by G.I. Zhuravlev, M.P. Sarafanova, Ye.K. Yerchenkova, and I.V. Borisenko, Moscow Oil and Gas Institute imeni I.M. Gubkin]

UDC 541.15:665.2.097.3

[Abstract] A number of researchers have examined the effect of different factors on the formation of a catalyst's active structure. In a continuation of this research, the authors of the study reported herein examined the effect that  $\gamma$ -irradiation of hydrogels of aluminosilicate in the different stages of its preparation exerts on the composition of the products of cracking light gas oil at a boiling temperature between 240 and 350°C. Laboratory specimens of aluminosilicate were synthesized by the method of coprecipitation of gel-forming solutions in accordance with a procedure that maintained all of the stages used when catalyst is synthesized commercially. The specimens were subjected to  $\gamma$ -irradiation in doses up to 50 and 100 kGy at dose rates of 23, 32, and 95 kGy/h on MRX-100 and K-60,000 units. The hydrogels were used to crack a kerosene gas oil fraction on a laboratory unit at 380°C while the raw material was fed through at a space velocity of 2.4 cm<sup>3</sup> raw material per cm<sup>3</sup> catalyst per hour. The group and individual compositions of the gasolines were determined by luminescent-chromatographic analysis and gas-liquid chromatography. Aromatic, olefin, and paraffin-naphthene hydrocarbon groups were identified. The content of olefin hydrocarbons was found to increase significantly as the radiation dose increased. The content of aromatics, on the other hand, remained virtually unchanged, and the content of paraffin-naphthene hydrocarbons decreased. The authors then proceeded to compare the group composition of cracking products produced on catalysts irradiated in an activation solution with specimens irradiated in catalyst rinse waters. Irradiation to a dose of 100 kGy in catalyst rinse waters was found to result in an increase in olefin hydrocarbons that was a factor of 1.3 higher (after boiling at 150°C) than in the case of irradiation to the same dose in an activation solution. The changes in the chemical composition of the cracking gasolines were attributed to the fact that irradiation of the catalyst in hydrogel rinse waters affects the formation of the active structure in such a way that the rate of the secondary reaction of the redistribution of hydrogen diminishes and the cracking products become enriched with olefin hydrocarbons. As has also been indicated elsewhere, the ratio of the Lewis and Bronsted acid centers in the aluminosilicate catalyst changes as a result of irradiation (i.e., the relative content of aprotinated centers increases in comparison with a nonirradiated catalyst specimen). All of this is determined to indicate

the complex nature of the interaction of  $^{60}\text{Co}$   $\gamma$ -radiation with the gel-activated solution or gel-rinse water system during the catalyst preparation process. Figure 1, table 1; references 7 (Russian).

### Radiochemical Transformations of Dimethylphenylsilsesquioxane Block Copolymers in a Vacuum

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[Article by V.V. Lyashevich, L.N. Pankratova, O.Yu. Panov, E.R. Klinshpont, A.Yu. Rabkina, and B.G. Zavin]

UDC 541.15.678.84

[Abstract] Polyorganosiloxanes are widely used as binders for producing heat- and radiation-resistant materials. Methods of synthesizing block copolymers that make it possible to change the number of dimethylsiloxane and phenylsilsesquioxane blocks in the macromolecule have recently been developed that make it possible to purposively vary the properties of the resultant polymers. The radiochemical transformations of such block copolymers have remained virtually unstudied, however. In view of this fact, the authors of the study reported herein studied the radiochemical transformations of organosilicon block copolymers with different contents of dimethylsiloxane and phenylsilsesquioxane blocks. They also studied the effect of the relative content and number of blocks on the radiation stability of block copolymers. Serving as study objects were block copolymers with the general formula  $[(\text{CH}_3)_2\text{SiO}]_m[(\text{C}_6\text{H}_5\text{SiO}_{1.5})_n[\text{C}_6\text{H}_5\text{SiO}(\text{OH})]_{1-n})_n$  with dimethylsiloxane-to-phenylsilsesquioxane ratios of 20:7 (specimen 1), 20:13 (specimen 2), 40:9 (specimen 3), and 40:22 (specimen 4) and with respective average molecular masses of 21,000, 17,900, 4,400, and 5,900. The radiochemical yields of macroradicals and gaseous destruction products and the cross links were determined. The phenylsilsesquioxane blocks were found to exert a stabilizing effect. It was also discovered that the size of the dimethylsiloxane block has a decisive effect on the block copolymer's stability, whereas the size of the phenylsilsesquioxane block exerts only an insignificant effect. The authors propose a diagram of the radical reactions involved and rank the specimens tested in radiation stability series. These series are as follows:  $G_{\text{cross links}}$ : specimen 1  $\approx$  specimen 2 > specimen 4 > specimen 3 and  $G_{\text{gas}}$ : specimen 2 > specimen 1 > specimen 4 > specimen 3. Figures 2, tables 4; references 19: 16 Russian, 3 Western.

### Mass Spectrometry of the Oligomer Products of the $\gamma$ -Irradiation of Epichlorhydrin

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[Article by V.N. Doroshenko, I.R. Yentinzon, V.A. Nazarenko, and V.A. Pokrovskiy, Surface Chemistry Institute, UkSSR Academy of Sciences]

UDC 543.51:541.15

[Abstract] The products of the radiochemical oligomerization of  $\alpha$ -oxides are formed in a limited amount and often with a low molecular mass. The mechanism of these radiochemical reactions is not yet fully understood, however. In view of this fact, the authors of the study reported herein used the method of field desorption mass spectrometry to establish the nature of the oligomer products of  $\gamma$ -irradiated epichlorhydrin and to determine chemical reactions corresponding to them. The epichlorhydrin specimens were purified, irradiated, and subjected to dosimetry analysis as described elsewhere. The epichlorhydrin specimen were subjected to  $^{60}\text{Co}$   $\gamma$ -irradiation in air and in a vacuum at absorbed doses of 0.05 to 1.2 MGy and a dose rate of 2.0 Gy/s. The content of viscous product, i.e., polyepichlorhydrin, was determined to range from 1 to 15% at absorbed doses of 0.05 to 1.0 MGy. The molecular mass of the polyepichlorhydrin, as determined by the cryoscopic method, did not exceed  $M_n = 400$ . A modified MI-1201 was used to obtain the mass spectra. A tungsten filament 10  $\mu\text{m}$  in diameter with gold microdendrites precipitated on it was used as the emitter. The error in determining the mass numbers amounted to  $\pm 0.5$  amu. The polyepichlorhydrin was dissolved in excess toluene and applied onto the field emitter's surface. The studies performed indicated that the oligomers represent a mixture of molecules of the type  $-(\text{CH}_2)_n\text{-CO-}$  ( $M_n \approx 400$ ) or  $-(\text{O-CH}_2\text{CH}(\text{CH}_2\text{Cl})_n\text{-})_n$  ( $M_n \approx 800$ ). These two types of molecules were determined to be formed in accordance with radical and ion mechanisms, respectively. Studies of the dependence of the polyepichlorhydrin yield on the concentration of radical, proton, and electron acceptors have shown that inhibitors of the ion and radical reaction cause a reduction in the polymer yield. Nitrobenzene (a radical acceptor in the amount of 1 mol/l and with an irradiation dose of 0.3 Gy) was thus found to reduce the epichlorhydrin yield from 7% (by weight) in a pure monomer up to 3% (by weight) with a simultaneous increase in  $M_n$  from 370 to 1,000 amu. Proton acceptors were found to be less effective, whereas methylenechloride was found to increase the yield of polymer to 11% and  $M_n$  to 1,100 amu. Figure 1; references 9: 7 Russian, 2 Western.

### Track Effects in the Nonstationary Radiant Electric Conduction of Polymers

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[Article by A.P. Tyutnev, S.G. Boyev, V.N. Abramov, Ye.A. Kuzmenkov, and A.V. Vannikov, High Voltages Scientific Research Institute, Tomsk Polytechnic Institute imeni S.M. Kirov, and Electrochemistry Institute, USSR Academy of Sciences]

UDC 541(15+64)

[Abstract] The authors of the study reported herein examined the nonstationary radiant electric conduction of 14 polymers subjected to pulsed (0.8 to 3.6 ms) irradiation by protons with an energy of 7.7 MeV and  $\alpha$ -particles with an energy of 20 MeV at room temperature in the stages of their rise and fall. The following polymers are studied: polystyrene, polyethylene naphthalate, polyethylene terephthalate, polypyromellitimide (Kapton), high-pressure polyethylene, polypropylene, polyoxadiazole, polytetrafluorethylene, polymethyl methacrylate, (chlorinated) polyvinylchloride, aromatic polyamide, polyvinylidene fluoride, polyvinylfluoride, and fluoroplastic 4MB. The studies performed represent a continuation of a systematic investigation of the nonstationary radiant electric conduction of polymers on which the authors have reported previously. The studies reported herein demonstrated that ionization efficiency decreases in a regular pattern upon the transition from fast electrons to protons and then to  $\alpha$ -particles. In the case of  $\alpha$ -particles, the decrease observed ranged from a factor of 10 to a factor of 23 in polymers with a free-charge mechanism of radiant electric conduction. The authors discuss the results of their experiments within the framework of a simplified structure of a track of charged particles. Figures 2, tables 2; references 11: 9 Russian, 2 Western.

**Transformation of Hexafluoropropylene in a Reduced-Pressure High-Frequency Discharge**

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[Article by G.K. Vinogradov, D.I. Slovestkiy, and A.G. Timokhov, Petrochemical Synthesis Institute imeni A.V. Topchiyev, USSR Academy of Sciences]

UDC 541:645:687.742

[Abstract] In view of the importance of transformations of organic compounds in electric discharge and the extensive use of fluorocarbons in creating coatings, the authors of the study reported herein studied the kinetic laws governing the formation of stable gas-phase products of the transformation of hexafluoropropylene under flowthrough conditions in a reduced-pressure high-frequency discharge. The studies were performed by using a flowthrough-type reactor with plane-parallel brass electrodes enclosed in a Teflon case and an electrostatic copper screen. The length and width of the interelectrode space corresponded to the sizes of the electrodes (120 x 60 mm), and the interelectrode gap amounted to 20 mm. Openings were evenly spaced every 15 mm along the reactor's side wall to permit sampling of the gas-phase products and monitor the evenness of the discharge. Hexafluoropropylene with a purity of 99.8% containing C<sub>4</sub> impurities was used for the experiments. The experiments yielded a wide range of C<sub>1</sub> through C<sub>8</sub> fluorocarbons. These were subjected to chromatographic and chromatographic-mass spectrometric analysis. Tetrafluoroethylene and saturated C<sub>1</sub> through C<sub>4</sub> fluorocarbons were the main products formed. Tetrafluoropropyne, hexafluorobutene-2, and traces of octafluorocyclobutane were also formed. Processes entailing the formation and participation of atomic fluorine were determined to play an important role in the reaction mechanism proposed by the authors and confirmed by spectroscopic measurements of its concentration in the discharge and by experiments involved fluorination of hexafluoropropylene. Analysis of processes involving charged particles showed that their rate was limited by the ionization rate (estimated at 1 to 5% of the transformation rate of hexafluoropropylene) and were therefore insignificant. Figures 2, table 1; references 15: 7 Russian, 8 Western.