"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

VODOP'YANOV, K.A.; VOROZHTSOV, B.I.; MIKHAYLOVA, T.G.

Effect of gamma radiation on the dielectric properties of some electric insulating materials. Izv. vys. ucheb. zav.; fiz. no.4:152-155 '60. (MIRA 13:9)

1. Sibirskiy fiziko-tekhnicheskiy institut pri Tomskom gosuniversitete imeni V.V. Kuybysheva.
(Gamma rays) (Electric insulators and insulation)

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VODOP'YANOV, K.A.; VOROZHTSOV, B.I.; OL'SHANSKAYA, N.I.

Effect of gamma radiation on the dielectric properties of some electric insulating materials. Part 4: Polyethylene. Izv. vys. ucheb. zav.p fiz. no.4:156-159 '60. (MIRA 13:9)

1. Sibirskiy fiziko-tekhnicheskiy institut pri Tomskom gosuniversitete im. V.V. Kuybysheva.
(Gamma rays) (Electric insulators and insulation)

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VODOP'YANOV, K.A., doktor tekhn.nauk, prof.; VOROZHTSOV, B.I., kand. fiz.-matem.nauk, dotsent; POTAKHOVA, G.I., kand.fiz.-matem. nauk; OLSHANSKAYA, N.I., inzh.

Electric and physical properties of electric insulation materials subjected to radiation. Elektrichestvo no.5:60-66 My '60. (MIRA 13:9)

1. Sibirskiy fiziko-teknicheskiy institut pri Tomskom Gosudarstvennom universitete.
(Electric insulators and insulation)
(Materials, Effect of radiation on)

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

85165

21.6600 (1403,1544,2209) S/139/60/000/005/020/031
E073/E135

AUTHORS: Vodop'yanov, K.A., and Vorozhtsov, B.I.

TITLE: Influence of Gamma-radiation on the Dielectric Properties of some Electrical Insulation Materials. V. Silicon-Organic Materials of the Rubber Type

PERIODICAL: Izvestiya vysshikh uchebnykh zavedeniy, Fizika, 1960, No. 5, pp 118-123

TEXT: Whilst the changes in the physical and chemical properties and structural changes caused by γ-radiation in various polymers have been studied, the changes in the electrophysical properties have not been studied to the same extent. All the investigated grades of material were produced from a polydimethylsiloxane elastomer and various fillers: 14p-2 (14r-2) - titanium dioxide, 14p-6 (14r-6) - silica gel and zinc whiteners, and 14p-15 (14r-15) - silica gel, zinc whiteners and red oxide. The specimens were exposed to radiation from a betatron Б-15 (B-15) under various temperature conditions. The strength of the radiation dose was 500 r/min: the measuring technique was the same as that used in other work of the authors (Ref. 10). The graph of Fig. 1 shows the frequency dependence of tg δ and of

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Influence of Gamma-radiation on the Dielectric Properties of Some Electrical Insulation Materials. V. Silicon-Organic Materials of the Rubber Type

the dielectric constant of the rubber 14r-2 before and after irradiation; tg δ increases appreciably as a result of irradiation whereby the difference increases with decreasing frequency. There is hardly any change at all in the dielectric constant as a result of irradiation. Figs 2, 3 and 4 show the temperature dependences of tg δ for the frequencies 50, 1000 and 10 000 c.p.s; it can be seen that with increasing temperature the loss angle increased both for the non-irradiated and irradiated specimens. It was found that the increase in electric conductivity of irradiated specimens at room temperature remains the same irrespective of the applied integral irradiation dose. Some of the specimens were only subjected to pressing without final baking at 200 °C; it was assumed that material which has been incompletely polymerized will react differently to radiation than ordinary material. It was found that in such specimens the loss angle decreased after irradiation instead of the increase

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S/139/60/000/005/020/031
E073/E135

Influence of Gamma-radiation on the Dielectric Properties of Some Electrical Insulation Materials. V. Silicon-Organic Materials of the Rubber Type

observed on the other specimens. Another interesting feature was that the dependence of the loss angle on the frequency in the irradiated incompletely polymerized material is the same as the analogous curve for the normal material. Thus, in addition to processes leading to an increase of the electric conductivity and the loss angle of normal material, irradiation also assists in completing the polymerization of the incompletely polymerized material to its final stage. This is in agreement with an observation made by R. Harrington (Ref. 6). Entirely different results were obtained for the rubber 14r-15, for which the dielectric losses have a clearly pronounced relaxation character (Figs 7-9); after irradiation the dependence of the loss angle on the frequency decreases. The greatest decrease was observed in the case of irradiation at +60 °C, whilst irradiation at -60 °C did not bring about any change in the frequency dependence of the loss angle. The temperature dependence of the dielectric loss angle measured in the frequency range 40-10^6 c.p.s. (Figs 8, 9)

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S/139/60/000/005/020/031
E073/E135

Influence of Gamma-radiation on the Dielectric Properties of Some Electrical Insulation Materials. V. Silicon-Organic Materials of the Rubber Type

for the material 14r-15 shows that as a result of irradiation the relaxation maxima shift towards higher temperatures; irradiation also leads to an increase in the mechanical strength.
Acknowledgements are made to A.I. Tovbin and V.D. Dedkov who participated in the experiments.

There are 9 figures, 1 table and 10 references: 7 English and 3 Soviet.

ASSOCIATION: Sibirskiy fiziko-tekhnicheskiy institut pri Tomskom gosuniversitete imeni V.V. Kuybysheva (Siberian Physics and Engineering Institute at Tomsk State University imeni V.V. Kuybyshev)

SUBMITTED: December 3, 1959

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

246820

80154
S/105/60/000/05/14/028
B007/B008

AUTHORS: Vodop'yanov, K.A., Professor, Doctor of Technical Sciences, Vorozhtsov, B.I., Docent, Candidate of Physical and Mathematical Sciences, Potakhova, G.I., Candidate of Physical and Mathematical Sciences, Ol'shanskaya, N.I., Engineer

TITLE: The Electrical and Physical Properties of Technical Electric Insulation Materials When Subjected to Radioactive Irradiation

PERIODICAL: Elektrichestvo, 1960, No. 5, pp. 60-66

TEXT: Experimental data are given in the paper under review. The influence of gamma radiation on the electrical and physical characteristics of highly polymeric dielectrics, siliconorganic and phenol formaldehyde synthetic materials. Irradiation was carried out with a betatron (design by the Tomskiy politekhnicheskiy institut (Tomsk Polytechnic Institute)) with an energy of the gamma rays of 15 Mev and a dosage rate of 300-1200 r/min. The samples were irradiated at various temperatures (-60, +20, +60°C) and at tropical humidity (+40°C and relative humidity of the air of 98%). The following was determined on the basis of these experiments described here in detail. High-polymer dielectrics of the

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The Electrical and Physical Properties of Technical Electric Insulation Materials When Subjected to Radioactive Irradiation

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B007/B008

polyethylene-, "Fluoroplast"-, and polystyrene type do not change the mechanism of the dielectric losses and the polarization at an irradiation with gamma rays of a dosage of up to 10^6 at room temperature. The absolute values of the dielectric constants, of the loss angle, and the electric strength remain unchanged. An increase of the electrical conductivity is observed in polyethylene on low-temperature irradiation, and a reduction on irradiation under tropical conditions. In the case of the "Steklotekstolit SKM-1" (organosilicon synthetic material), the greatest changes occur as a result of low-temperature irradiation and at tropical humidity. The loss angle and the dielectric constant change most in consequence of the irradiation in the range of low frequencies. The electrical conductivity and the ohmic part of the dielectric losses increase in organosilicon rubber after irradiation. An irradiation of raw rubber with gamma rays accelerates the vulcanizing process. An increase of the losses is observed at an irradiation with gamma rays of the phenol formaldehyde synthetic materials. The loss angle changes most after an irradiation at low temperatures and under tropical conditions. The dielectric constant and the electric strength of these synthetic materials do not change after an

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80154

The Electrical and Physical Properties of Technical Electric Insulation Materials When Subjected to Radioactive Irradiation

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B007/B008

irradiation with gamma rays under the conditions mentioned. It is stated on the basis of these experimental data that the radiation strength of electric insulation materials depends on the physical and chemical properties of the material and its structure, as well as on the exterior conditions during irradiation and investigation. The authors are of the opinion that the character of the polarization and the dielectric losses in one or the other temperature- and frequency range is the most important criterion for the radiation strength of the dielectric. It is necessary to know the temperature- and frequency characteristics of the dielectric constant and the angle of dielectric losses before expressing an opinion on the relative stability of the electric insulation material. The following persons took an active part in the experiments: V.D. Dedkov, Ye.A. Zimina, M.D. Lavrov, T.G. Mikhaylova, Ye.S. Nesmelova, T.B. Nedokos, L.A. Prudnikova, G.V. Sitozhevskaya and A.I. Tovbina. There are 16 figures.

ASSOCIATION: Sibirskiy fiziko-tekhnicheskiy institut pri Tomskom Gosudarstvennom universitete (Siberian Physics and Technology Institute at the Tomsk State University)

SUBMITTED: December 7, 1959

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

S/139/62/000/006/021/032
E194/E155

AUTHORS: Vorozhtsov, B.I., Potakhova, G.I., and Nesterov, V.M.

TITLE: Dielectric properties of insulating materials during gamma radiation. III. Plastic АГ-4 (AG-4)

PERIODICAL: Izvestiya vysshikh uchebnykh zavedeniy, Fizika, no.6, 1962, 143-146

TEXT: Until recently, plastic grade AG-4, which is based on phenol-formaldehyde, was considered one of the few heat-resistant moulded materials suitable for casings for capacitors, transformers, relays etc. operating at temperatures of +200 °C. It is becoming increasingly necessary to test such electrical and radio materials environmentally. The present work describes the study of the permittivity, tan δ, breakdown stress and resistivity of plastic grade AG-4 under gamma radiation from Co^{60}, at a dosage rate of 670 rads/min with total dosages up to 10^5 rads, at various temperatures between -60 and +200 °C, in the frequency range 30 - 10^5 c/s. The permittivity changed less than 10%. A dosage of 2000 rads/min and a total dose of up to 4×10^5 rads/min had no effect on the electric strength at 50 c/s. The tan δ changed

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

Dielectric properties of insulating... S/139/62/000/006/021/032
E194/E155

irreversibly, particularly at high temperatures and low frequencies (at a frequency of 30 c/s and T = 200 °C the change was 50%). In the temperature and frequency range in which relaxation losses occur in plastic AG-4, reversible changes are observed in the nature of the frequency function of tan δ, presumably due to displacement of the maximum of tan δ towards lower frequencies because of the extended range of relaxation time whilst subject to radiation.
There are 5 figures.

ASSOCIATION: Sibirskiy fiziko-tekhnicheskiy institut pri Tomskom gosuniversitete imeni V.V. Kuybysheva (Siberian Physicotechnical Institute at Tomsk State University imeni V.V. Kuybyshev)

SUBMITTED: December 18, 1961

Card 2/2

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

VODOP'YANOV, K.A.; VOROZHTSOV, B.I.

On the question of the measurement of the dielectric loss angle and permittivity of solid dielectrics in the regions of ultra-short and decimeter waves. Zh.tekh. Fiz. 22, No.11, 1877-80 '52. (MLRA 5:12)
(PA 56 no.668:5461 '53)

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

VOROZHTSOV, B.I.

VOROZHTSOV, B.I.--"Electrical Properties of Fused Quartz in the Presence of High Frequencies and High Temperatures." *(Disseratations For Degrees in Science And Engineering Defended At USSR Higher Educational Institutions). (34). Tomsk State U imeni V.V. Kuybyshev, Tomsk, 1955.

SO: Knizhnaya Letopis' No. 34, 20 August 1955

* For the Degree of Candidate in Physicomathematical Sciences

APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

ACCESSION NR: AP4041843 S/0139/64/000/003/0007/0011

AUTHORS: Vorozhtsov, B. I.; Filatov, I. S.

TITLE: Effect of gamma radiation on the dielectric properties of a vacuum dense ceramic

SOURCE: IVUZ. Fizika, no. 3, 1964, 7-11

TOPIC TAGS: ceramic dielectric, ceramic technology, gamma radiation, dielectric constant, dielectric loss

ABSTRACT: In studying a talc-base ceramic, the authors plotted the dependence of the tangent of the dielectric loss angle and the dielectric constant on the temperature, frequency, and field intensity before, during, and after irradiation with gamma rays from Co^{60} at 20C and at relative humidity 68%. It is concluded that the properties of the ceramic, both following the action of gamma radiation and during the interval of γ-irradiation in a weak field, can be noticeably changed (as a result of destruction of the structure of the material following vitrification with formation of color centers) only when the radiation dose is large (more than 10^6 r). The relaxation losses decrease with increasing dose

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ACCESSION NR: AP4041843

and the conductivity losses increase somewhat. In strong fields, even a slight radiation dose (2.25×10^4 r) leads to a sharp increase in the dielectric constant and loss angle, owing to the strong dependence of the ionization of the gas in the pores of the material in the hf field on the action of the external ionizer. Orig. art. has: 5 figures.

ASSOCIATION: Sibirskiy fiziko-tekhnicheskiy institut pri Tomskom gosuniversitete imeni V. V. Kuyby*sheva (Siberian Physicotechnical Institute at Tomsk State University)

SUBMITTED: 25Jul62 ENCL: 02 SUB CODE: EC, SS

NO REF SOV: 013 OTHER: 006

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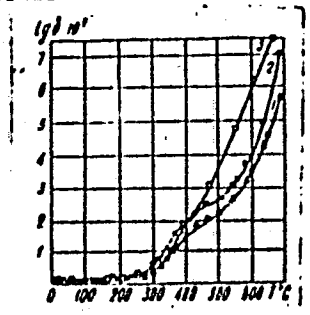
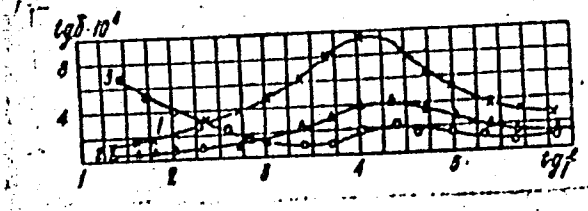
ACCESSION NR: AP4041843

ENCLOSURE: 01



Temperature dependence of loss angle befire abd after irradiation. Curve 1 - strong field, Δ - before irrad. o - after irradiation. Curve 2 - strong field, o - before irradiation ×, □ - after irradiation



Frequency dependence of ceramic loss angle before and after irradiation.
1 - before irradiation
2 and 3 - after irradiation

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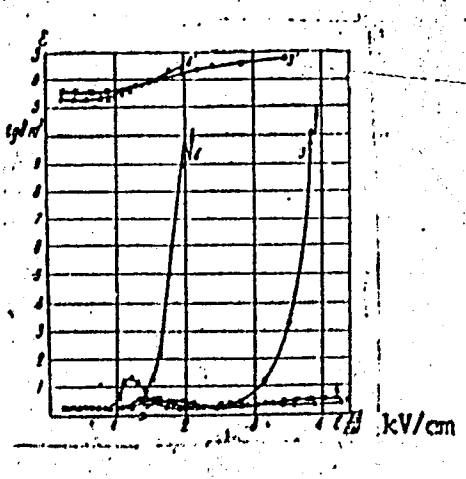
ACCESSION NR: AP4041843

ENCLOSURE: 02



Dependence of loss angle and dielectric constant of a ceramic on the field intensity at the instant of irradiation.

1, 2 - before irradiation
3, 3', 4, 4' - after irradiation

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

SOV/112-58-2-1871

Translation from: Referativnyy zhurnal, Elektrotekhnika, 1958, Nr 2, p 12 (USSR)

AUTHOR: Vorozhtsov, B. I.

TITLE: Some Electrical Characteristics of Fused Quartz at High Frequencies, High Voltages, and High Temperatures (Nekotoryye elektricheskiye svoystva plavlenogo kvartsa pri vysokikh chastotakh, povyshennykh napryazheniyakh i vysokikh temperaturakh)

PERIODICAL: Izv. Tomskogo politekhn. in-ta, 1956, Vol 91, pp 363-376

ABSTRACT: Investigations of temperature and frequency effects on tgδ by means of "air" electrodes were carried out with tubular specimens made from three grades of fused quartz. Effect of temperature on tgδ of fused quartz could be subdivided into two sections: low-temperature (up to 500° C) where tgδ is practically independent of temperature and very small in its absolute value; and high-temperature where tgδ is sharply dependent on temperature. At high temperatures, tgδ is inversely proportional to frequency; measured tgδ is equal to that calculated on the basis of conductance; high-frequency heating and

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SOV/112-58-2-1871

Some Electrical Characteristics of Fused Quartz at High Frequencies, High . . . .

DC heating at the same voltage on the specimen are also equal. The high-temperature part of tgδ in the fused quartz is determined by the through conductance. A thermal treatment at 1,100°-1,300° C for 2-3 hours lowers the tgδ by 40-50%. A reduction in tgδ is also observed as a result of electrical cleaning of specimens. Thus, fixing a part of impurity ions during calcination or removal of them during the electric cleaning results in improved electric insulation characteristics of fused quartz. Dielectric losses in the specimens having gas inclusions consist of a conductance loss and an ionization loss. The latter are responsible for the high value of tgδ of these specimens, dependence of tgδ on voltage, and a steeper increase of tgδ with temperature.
Bibliography: 6 items. Sibirskiy fiziko-tekhnich. in-t (Siberian Physics-and-Technology Institute), Tomsk.

M.D.M.

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

SOV/139-58-4-17/30

AUTHORS: Vorozhtsov, B. I. and Filatov, I. S.

TITLE: Non-Steady State Calorimetric Method of Measuring the Dielectric Loss Angle and the Permittivity in Strong High Frequency Fields (Nestatsionarnyy kalorimetricheskiy metod izmereniya ugla dielektricheskikh poter' i dielektricheskoy pronitsayemosti v sil'nykh polyakh vysokoy chastoty)

PERIODICAL: Izvestiya Vysshikh Uchebnykh Zavedeniy, Fizika, 1958, Nr 4, pp 105-113 (USSR)

ABSTRACT: Calculation of the dielectric loss angle is based on comparing the heat generated by the dielectric losses and the heat measured from the temperature rise of the dielectric, taking into consideration the heat losses due to heat exchange between the specimen and the surrounding medium. The mathematical formulation of this relation is expressed by the differential equation (2), p 106, the solution of which can be developed in series, the author limits himself to using the first term, thus obtaining the following equation:

$$\Delta T = (A/m\lambda)t$$

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Non-Steady State Calorimetric Method of Measuring the Dielectric Loss Angle and the Permittivity in Strong High Frequency Fields

In the case of non-uniform fields, the relations are more complicated. Of practical interest is the case of a cylindrical condenser and the solution of the non-uniform equation of heat transfer for such a case, quoted from an earlier paper of one of the authors (Ref 16), is expressed by Eq.(8). The equation on which the calculation of the dielectric losses is based is:

$$\operatorname{tg} \delta = \frac{m\lambda \Delta T}{0.24\ \omega\ CV^2_{rms} t}$$

The block schematics of the test set-up is shown in Fig.2; it consists of an H.F. oscillator, an inductively coupled metering circuit, equipment for measuring the temperature of the specimen and its heating by the high frequency current, metering voltage and a crucible electric furnace. The oscillator can be operated at frequencies between 7.65 and 85 Mc/sec. All the connections in the oscillator and in the meter circuit were made of copper tubes and silver-plated; coils intended

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

SOV/139-58-4-17/30

Non-Steady State Calorimetric Method of Measuring the Dielectric Loss Angle and the Permittivity in Strong High Frequency Fields

for operation at frequencies above 20.5 Mc/sec are also silvered. The metering circuit consists of an induction coil, a tuning condenser C_0, a condenser containing the dielectric, C_x and a voltage metering circuit. The error of determining the capacitance is 0.5% with a sensitivity of 0.025 pF per scale division. The temperature dependence ε was calculated from the change in the capacitance with temperature and the geometrical dimensions of the specimen with an error of 1.5%; it can be seen from the graph, Fig.3, that for quartz the thus obtained results are fully in agreement with those measured on a Q-meter. In a table, p 112, a comparison is given of the dielectric loss factor for several materials measured by the here described method with the values measured by other methods. The here described non-steady state calorimetric method of measuring the dielectric characteristics of solid dielectrics in strong fields enables measuring at temperatures up to 1000°C and even higher and determining the dependence of the dielectric loss factor on the field potential at various

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SOV/139-58-4-17/30

Non-Steady State Calorimetric Method of Measuring the Dielectric Loss Angle and the Permittivity in Strong High Frequency Fields

temperatures and frequencies. The test equipment is simple in design and the test procedure itself does not differ greatly from that applied in current methods of measuring the dielectric characteristics.
There are 6 figures, 1 table and 19 references, 11 of which are Soviet, 3 German and 5 English.

ASSOCIATION: Sibirskiy fiziko-tekhnicheskiy institut pri Tomskom gosuniversitete imeni V. V. Kuybysheva (Siberian Physico-Technical Institute at the Tomsk State University imeni V. V. Kuybyshev)

SUBMITTED: January 31, 1958

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APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0"

"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

VOROZHTSOV, B.I.

AUTHOR: Kuchin, V. D., Candidate of Technical Sciences SOV/105-58-7-23/32

TITLE: Conference on Solid Dielectrics and Semiconductors (Konferentsiya po tverdym dielektrikam i poluprovodnikam)

PERIODICAL: Elektrichestvo, 1958, Nr 7, pp. 84 - 85 (USSR)

ABSTRACT: The conference took place from February 3rd to February 8th, 1958, in the Tomsk Polytechnical Institute, Section of Polarization, Losses, and Conductivity. B.I.Vorozhtsov, Candidate of Technical-Mathematical Sciences (SFTI) spoke about a simple circuit for the measurement of tg δ of from $1 \cdot 10^{-4}$ to 1 and of the capacitance of from 5 to 20 pF. The accuracy of measurement was 10% in the case of tg δ and 5% in the case of the capacity. Professor G.A.Smolenskiy and others (Institute of Semiconductors AS USSR) ~~investigated~~ "the possibility of obtaining multicomponent systems from oxygen oxides without investigation of the state diagrams of the latter." I.S. Filatov (SFTI) "Investigated" tg δ and ε of several ceramic materials in fields of from 180 - 4600 V/cm in the frequency range of from 7,65 - 20,5 megacycles and in the temperature range of from

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20 - 720°C according to the calorimetric method. Ionization-, relaxation-, and conductivity losses were found to occur. V.M. Nesterov (SFTI) measured by means of the wave guide method the conductivity, the dielectric permeability, and the losses of the "fluoroplast", polyethylene, "vinyloplast" etc. at from -80 - +100°C and 10^6, 10^7, 5.10^8 and 3.10^9 cycles. S.E.Ryabochenko, Irkutsk University (Irkutsk University) in the range of from 20 to 140°C found two temperature maxima of tg δ of the alkali halide crystals which are connected with the hydroscopy of the latter. I.G.Vorozhtsova (SFTI) found relaxation maxima in muscovite. They are assumed to be connected with the peculiarity of structure. Docent I.P.Tonkonogov and Ye.T.Nadirov (Karaganda Mining Institute) investigated the magnetic and electric heating of coals on the basis of the Maxwell-Wagner model. Docent Ye.K.Zavadovskaya (TPI) found that the character of the dependence of the electric conductivity of solid solutions on the composition in the case of temperature variation is determined by the interaction forces of the ions in the crystal. Docent K.N.Pogodayev and V.G.Stepanchenko (Irkutsk University) in the range of from 50 to 300°C found temperature maxima of the direct and reversible currents in natural nickel

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or in nickel activated by ions or in the copper of rock salt. V.I.Gaman (SFTI) found that the temperature course of the pool factor in the case of silicate- and boron silicate glasses is to a considerable extent determined by the temperature dependence of the polarization potential in the case of the existence of a high-voltage polarization. Professor F.I.Kolomoytsev and A.Ya. Yakunin, Dnepropetrovsk University (Dnepropetrovskiy universitet) found the variation of the electric conductivity and the EMF induced by X-ray radiation by temperature and the intensity of the X-rays in polyethylene, polystyrene, teflon, polymethylmethacrylate, mica, and other dielectrics. Docent Ya.N.Pershits (Pskov Institute of Pedagogics) and G.D.Tarasenko (North Ossetian Institute of Pedagogics, Ordzhonikidze) found that the character of the propagation of the F-center cloudlet and that of its mobility is changed in consequence of the primary coloration of the NaCl-, KCL-, KBr- and KJ crystals, whereas the electric conductivity of the ions of the samples is reduced irreversibly. N.M.Torbin (TPI) found that the X-ray absorption factor increases with increasing molecular weight of pressed alkali halide salts.

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Cobference on Solid Dielectrics and Semiconductors SOV/105-58-7-23/32

ASSOCIATION: Tomskiy politekhnicheskiy institut (Tomsk Polytechnical Institute)

1. Dielectrics--USSR 2. Semiconductors--USSR 3. Conferences

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SOV/120-59-1-27/50

AUTHOR: Vorozhtsov, B. I.

TITLE: Measurement of Dielectric Characteristics of Electrically Insulating Materials (Izmereniye dielektricheskikh kharakteristik elektroizolyatsionnykh materialov)

PERIODICAL: Pribory i tekhnika eksperimenta, 1959, Nr 1, pp 112-115 (USSR)

ABSTRACT: At the present time the dielectric constant and the dielectric loss angle of electrically insulating materials is usually measured by balanced bridge methods. These methods are very sensitive and accurate but involve a large number of operations leading to the final balance. Many years ago Moullin (Ref 1) proposed an unbalanced bridge which is shown in Fig 1. In this figure r_1 and r_2 are two equal non-reactive resistors, one of the capacitors is a variable air capacitor and the other contains the specimen under investigation. When the two capacitors are equal the current through the galvanometer is a minimum and is due to dielectric losses in the insulating material under investigation. If the input impedance of the indicator is very high then the loss angle is given by

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Measurement of Dielectric Characteristics of Electrically Insulating Materials

$\tan \delta_x = 4V_g/V$ where V_g is the voltage across the indicator and V is the input voltage. Moullin's bridge was modified by Mikhaylov (Ref 2) and Karandeyev (Ref 3) so that its sensitivity is increased and $\tan \delta_{min}$ was of the order of 3×10^{-4}. The circuit shown in Fig 2 which is a differential circuit is much simpler in practice. It is shown that:

$$\tan \delta = \frac{1}{R\omega C_x} = \frac{4V_g}{V} \sqrt{1 + \left(\frac{1}{2\omega C Z_g} + \frac{1}{2} \tan \delta\right)^2} \qquad (4)$$

The actual circuit used by the present authors is shown in Fig 3. The bridge was supplied by an audio-frequency oscillator with a symmetric output of up to a few hundred volts. The bridge indicator was a millivolt meter with a high input impedance, and the variable element was in the form of standard air capacitor. The millivolt meter is shown in Fig 4 and is based on a single pentode used as a cathode follower.

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SOV/120-59-1-27/50

Measurement of Dielectric Characteristics of Electrically Insulating Materials

The bridge may be used to measure dielectric losses and dielectric constants in the range 20-200 000 c/s. The value of tan δ may be in the range 10^{-4}-1 or more; the capacitance of the capacitor containing the dielectric may be between 7 and 250 pF. The main errors at room temperature are as follows: error in tan δ is $\pm(10\% + 5 \times 10^{-5})$ at 100 V while the error in the capacitance is $\pm(0.5\% + 1$ pF). There are 4 figures and 2 tables, 2 Soviet and 1 English reference.

ASSOCIATION: Sibirskiy fiziko-tekhnicheskiy nauchno-issledovatel'skiy institut (Siberian Physical Technical Scientific Research Institute)

SUBMITTED: September 27, 1957.

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PROCESSES AND PROPERTIES INDEX

Preparing phenol from chlorophenol. N. N. Vinogradov, Jr. and P. V. Karlash. Russ. 30,680, Aug. 31, 1933. ClC_6H_4OH is heated under pressure with water and Cu_2O to a temp. of 250-280°.

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CA

10

Preparing phenol from dichlorobenzene. N. N. Vorozhtsov, Jr. and P. V. Karlash. Russ. 30,000, Aug. 31, 1933. $C_6H_4Cl_2$ is heated to 350-380° under pressure together with H_2O and Cu_2O in the presence or absence of substances, such as $Ca(OH)_2$, combining with acid

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PROCESSES AND PROPERTIES INDEX

A-3

Reaction of o- and p-dichlorobenzene with cuprous oxide and water. N. N. Vorozhtsov and P. S. Karlason (Compt. rend. Acad. Sci. U.R.S.S., 1933, 222, 1-Oct) warming [illegible] $p\text{-}C_6H_4Cl_2$ with Cu_2O and H_2O at 275°/80 atm. only $PhOH$ is obtained. The reaction proceeds through $p\text{-}C_6H_4Cl\text{-}OH$. Cu_2O is a sp. reagent for this reaction. W. R. A.

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

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Co

10

Removing iron from ethylenediamine hydrochloride. N. N. Vorozhtsov, Jr. and V. A. Kobelev. Russ. 32,495, Oct. 31, 1933. Fe obtained in the interaction of dichloroethane with NH_4OH in steel autoclaves is removed by treatment with O_2 or with gases contg. O_2 such as air.

COMMON ELEMENTS

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

Ca

10

Thiophenol. N. N. Vorozhtzov, Jr., and S. F. Mishchengendler. Russ. 33,151, Nov. 30, 1933. PhSH is prepd. by heating Ph_2S under pressure, for example, in an autoclave or tube still at 300-380° with an aq. soln. of Na_2S.

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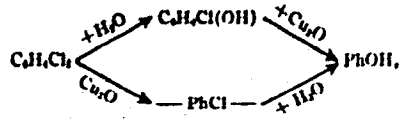
3RD AND 4TH ORDERS

10

The interaction of o- and p-dichlorobenzene with cuprous oxide and water. N. N. Vorozhtsov, Jr. and P. V. Karlash. *Compt. rend. acad. sci. U. R. S. S.* [N. S.], 1933, 221-3.—In the investigation of the possible development of a method for prepg. p-chlorophenol through the action of alkalies on p-$C_6H_4Cl_2$ in a Cu-lined autoclave the formation of phenol as a by-product was observed. This was caused by the reducing action of Cu_2O formed from metallic Cu through the influence of alkalies. The reaction was explained as follows:



because in heating p-$C_6H_4Cl_2$ with 1 mol. Cu_2O and H_2O only phenol is formed with a yield of 50% of the theory. The reaction takes 3 hrs. at 275° and 80 atm., yielding small amts. of PhCl (up to 10% of the theory). The reaction appears to take place via the formation of p-chlorophenol as an intermediate product, because phenol is also obtained when heating p-chlorophenol with Cu_2O. The phenol yield is unchanged when using o-$C_6H_4Cl_2$ which

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contains some p-$C_6H_4Cl_2$. Only small amts. of phenol were detected when using other reducing media instead of Cu_2O. The Cu_2O is a sp. reagent which replaces one Cl through H in the $C_6H_4Cl_2$ and the other through OH, acting simultaneously as a reducing agent and as a base.

A. A. Bochtlingk

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Ca

Reaction of chlorobenzene with sodium sulfide and sodium hydrosulfide. N. N. Vorozhtsov, Jr. and S. P. Mitzengendler. *Compt. rend. acad. sci. (U. S. S. R.)* [N. S.], 1933, 291-4(in German 294-5).—On heating PhCl with 0.5 mol. of 8% aq. Na_2S at 350-60° and 180-90 atm. 71.8-2.3% of the theoretical yield of Ph_2S and 15.6-15.8% of a mixt. of PhSH and PhOH is formed. With 6 mols. of Na_2S under the same conditions 2.8-5.5% Ph_2S and 84-92% of a mixt. of PhSH and PhOH is formed. The latter are formed thus: $Ph_2S + Na_2S = 2\ PhSNa$; $Ph_2S + 2\ NaOH = PhSNa + PhONa + H_2O$ from the previously formed Ph_2S. Similar results are had with NaSH.

Howard A. Smith

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CA

1ST AND 2ND ORDERS

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3RD AND 4TH ORDERS

10

Production of phenol from chlorobenzene. N. N. Vorochtzov, Jr., and A. G. Oshuev. *Anilinokrasochnaya Prom.* 3, 245-52(1933). The foreign literature is reviewed.

Chas. Blanc

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10

CA

Thiophenol. N. N. Vorozhtsov, Jr. and S. F. Mitsengendler. Russ. 34,554, Feb. 28, 1934. PhSH is prepd. by heating PhCl under pressure at 300-80° with an aq. soln. of Na$_2$S.

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3RD AND 4TH ORDERS

Ca

10

8-Hydroxyquinoline. N. N. Vorozhtzov, Jr., and S. F. Mitzengendler. Russ. 38,152, Aug. 31, 1931. 8-Chloroquinoline is heated to 200-300° with aq. solns. of alkalies in an autoclave under pressure and in the presence of metallic Cu or its compds. The product is sepd. from the reaction mixt. in the usual manner.

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12

Kinetics and mechanism of the catalytic exchange of chlorine for the amino group. N. N. Vorochtsov, Jr. and V. A. Kobelev. *Compt. rend. acad. sci. U. R. S. S.* 3, 108-11(in English 111-14)(1934).—In the presence of CuCl, PhCl reacts rapidly with NH_3 at 200° to give first an intermediate product $PhCl.Cu(NH_3)_2$ (I). I then reacts with NH_3, OH^- and $PhNH_2$ to form $PhNH_2$, PhOH and Ph_2NH, resp. It is not the concn. of NH_3 that dets. the reaction rate, but only the concn. of both PhCl and the catalyst that combine in the complex I. W. J. P.

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PROCESSES AND PROPERTIES INDEX

Sulfonation reaction. N. N. Vorozhtsev, Jr. *Anilinokrasitel'naya Prom.* 4, 84-7(1934).—A critical discussion of some of the conclusions of Ioffe (*C. A.* 28, 957[1], 1803[1]) on the kinetics of sulfonation reactions led to the following observations. The "const." of the equil. of sulfonation, as well as the consts. of the velocities of sulfonation and hydrolysis, is not a permanent value and is a function of the concn. of H_2SO_4 and the sulfonic acid. The magnitude of the "const." of equil. decreases with the decreasing concn. of H_2SO_4. The formula of v-sulfonation of Garrot and Courtot (cf. *C. A.* 24, 2439) is practically workable but theoretically incorrect. The correlation of isomers in the moment of equil. is a function of the concns. of H_2SO_4 and the sulfonic acids. Chas. Blanc

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Determination of phenol in the melt. N. N. Vorobiev, Jr. *Anilinokrasochnaya Prom.* 4, 166-7(1933). The method of Shemyakin (*C. A.* 28, 1305[5]) is time-consuming. The following rapid procedure produced accurate results with but slightly revivified melts. To 10 cc. of phenol melt (15-30 g. per l.) add in the cold 10 cc. 10% $BaCl_2$, filter, dil. the filtrate to a definite vol., withdraw 10 cc. of the soln. and titrate with Br_2 by the Koppeschaar method.

Chas. Blanc

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3RD AND 4TH ORDERS

C-O

10

The replacement of aromatic chlorine by the amino group. I. The preparation of aniline from chlorobenzene. N. N. Vorozhtsov, Jr., and V. A. Kobelev. *J. Gen. Chem.* (U. S. S. R.) 4, 310-23(1934); *cf. C. A.* 28, 6707[1].—$PhNH_2$ was obtained in 90% yield by heating PhCl with 5 mols. of concd. NH_4OH in the presence of 0.2 mol. Cu_2O at 200-300° under a pressure of 70 atm. for 2-3 hrs. PhOH (5%) and 1.5% of Ph_2NH were also formed. II. The preparation of *p*-nitroaniline from *p*-nitrochlorobenzene with copper compounds as catalysts. N. N. Vorozhtsov, Jr., and M. I. Krutkova. *Ibid.* 324-7.—Bivalent Cu compds. accelerate the conversion of *p*-$NO_2C_6H_4Cl$ to *p*-$NO_2C_6H_4NH_2$. Univalent Cu compds. and metallic Cu cannot be used because of their reducing action.

Lewis W. Butz

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1ST AND 2ND ORDERS

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3RD AND 4TH ORDERS

BC

B-II-1

Preparation of N.-W. acid from 1-chloronaphthalene. N. N. Vorozhcov and P. V. Karlasch (Anilinokras. Proiz., 1934, 4, 545—550).—α-Naphthol-4-sulphonic acid is obtained in theoretical yield by heating 1-chloronaphthalene-4-sulphonic acid with 5% aq. NaOH (3—4 mols.) at 200°/11—13 atm. for 7 hr., at 225°/23—24 atm. for 1·5 hr., or at 250°/38—40 atm. for 15 min. R. T.

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Production of aromatic amines from chlorine derivatives. N. N. Vorozhtsov, Jr. *Anilinokrasochnaya Prom.* 4, 232-40(1934).—In the 1st phase of interaction of PhCl (or other acyl chloride) with aq. NH_3 and a Cu salt PhCl (or other acyl chloride) reacts with the complex $Cu(NH_3)_2^+$ formed with the production of the addn. product $PhCl.Cu(NH_3)_2^+$ (1) in which the Cl is very mobile, reacting rapidly with NH_3, OH ion and $PhNH_2$ with the formation of $PhNH_2$, PhOH and Ph_2NH, resp., thus $PhCl.Cu(NH_3)_2^+ + NH_3 = PhCl.Cu(NH_3)_3^+$ (2); $PhNH_2 + HCl + Cu(NH_3)_2^+$ (3); $PhCl.Cu(NH_3)_3^+ + OH^- = PhOH + Cl^- + Cu(NH_3)_2^+$ (3); $PhCl.Cu(NH_3)_3^+ + PhNH_2 = Ph_2NH + HCl + Cu(NH_3)_2^+$ (4). The velocities of (2), (3) and (4) are so much greater that that of (1), that (1) alone dets. the over-all velocity. Therefore the rate of consumption of PhCl is independent of the concn. of NH_3, contrary to Groggins (*C. A.* 23, 3323; Groggins, *et al.*, *C. A.* 25, 1212; 26, 1344). The reaction velocity is $dx/dt = K(a - x)b$ (5) in which a is the initial concn. of PhCl and b the concn. of catalyst, which is const. The velocity of (2) is $d[PhNH_2]/dt = K_1c_0[NH_3]$, where c_0 is the concn. of $PhCl.Cu(NH_3)_2^+$. The velocity of (3) is $d[PhOH]/dt = K_2c_0(OH^-)$. The 2 equations integrated from 0 to t and the results divided give $[PhNH_2]/[PhOH] = (K_1/K_2) \times [NH_3]/[OH^-]$. Analogously the concn. of $PhNH_2$ dets. the amt. of PhOH formed according to (4). On increasing the concn. of NH_3 from 20 to 30% formation of $PhNH_2$ increases and that of PhOH decreases because the concns. of OH ions in 20 and 30% NH_3 are practically equal. Addn. of NH_4Cl to the reaction mixt. decreases the PhOH formation because of the resulting decrease of OH-ion concn., and retardation of the reaction rate (cf. U. S. pat. 1,607,924, *C. A.* 21, 249). Expts. show that when the autoclave is more nearly filled the reaction proceeds more rapidly and yields more $PhNH_2$ and less PhOH and Ph_2NH. The proposed reaction mechanism was confirmed by study of the interaction of PhCl and NH_3. The reaction was accomplished in a revolving autoclave set in an oil bath, the temp. of which could be raised to a desired point or cooled in 3-4 min. The runs were made at 200° with a large excess of NH_3 (33 mols. of NH_3 to 1 mol. of PhCl) and CuCl catalyst. The PhCl consumed was detd. from Cl^- formed in the reaction. The reaction-velocity const. was calcd. from the percentage of PhCl consumed in a unit of time. The reaction velocity is directly proportional to the concn. of catalyst. At concns. of 0.56 g.-mol./l. of PhCl and less the reaction velocity is directly proportional to the concn. of PhCl. At higher concns. of PhCl no such regularity was observed, because the excess of PhCl is insol. in aq. NH_3. The reaction is 0 order; its velocity at 200° is 4.7-5.1 × 10^3 b 2 g.-mol. PhCl/l. min., where b is the concn. of CuCl. The reaction velocity is practically independent of the concn. of NH_3 between 20 and 27%. At higher and lower concns. of NH_3 the velocity decreases, probably because of secondary factors. The velocity const. decreases as the reaction proceeds because the NH_4Cl produced lowers the reaction velocity. On the assumption that the concn. of the catalyst $Cu(NH_3)_2^+$ decreases in direct proportion to the amt. of NH_4Cl formed, the reaction velocity is $dx/dt = K(a - x)(b - nx)$, where n is the factor of proportionality. Integration leads to $K = [1/t(b - na)][\ln[a(b - nx)/(a - x)b]$. By solving graphically, $n = 0.02$. Study of the reaction with the addn. of NH_4Cl produced values for K and results substantially identical.

Chas. Blanc

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CA

10

Production of Neville and Winther acid from α-chloronaphthalene. N. N. Vorozhtsov, Jr., and P. V. Karlash. *Anilinokrasochnaya Prom.* 4, 545-50(1934).—α-$C_{10}H_7Cl$ (I), d_{15} 1.202, b. 255-65°, was obtained from Cl_2 and $C_{10}H_8$ in C_6H_6 (Traubenberg and Wasserman, *C. A.* 28, 601). A yield of 84% 1,4-$ClC_{10}H_6SO_3Na$ (II) resulted when to 60 g. I at 6° was added dropwise with stirring 92 g. of 100% H_2SO_4 at 10-12°, the reaction mass worked up with 200-300 cc. of satd. NaCl soln. and then steam-distd. from unchanged I (6%), sepd. from the 1,6-isomer by extn. with C_6H_6, concd., filtered after 12 hrs. and dried at 110°. By recrysta. from H_2O with addn. of a little NaCl resulted 50.2% II. A yield of 81% 1,4-$HOC_{10}H_6SO_3H$, m. 137°, was obtained by autoclaving 1 part of II with 4 parts of NaOH at concns. up to 10%. Poor results were obtained with higher concns. of NaOH (20-5%) (Eler, Ger. pat. 77,446; Ferrero and Bolliger, *C. A.* 23, 1896). With 5% NaOH the reaction is completed in 7 hrs. at 200° and 11-13 atm., in 1.5 hrs. at 225° and 23-4 atm. and in 15 min. at 250° and 38-40 atm. The reaction is accelerated by working in a Cu autoclave.

Chas. Blanc

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1st AND 2nd CODES

PROCESSES AND PROPERTIES INDEX

3rd AND 4TH CODES

11E

Ca

1 Vitamin C and narcotine. N. N. Vorobiev, Jr. and A. T. Troshtzenko. *Bull. Applied Botany, Genetics Plant Breeding* (U. S. S. R.), Suppl. 67, 161-4(1934).—A prepn. obtained from narcotine by demethylating it with HCl and heating showed no vitamin C activity.
J. S. Joffe

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CA

11E

The synthesis of vitamin C. N. N. Vorozhtsov, Jr. *Bull. Applied Botany, Genetics Plant Breeding* (U.S.S.R.) R.), Suppl. 67, 171-8(1934).—A review. J. S. Joffe

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3RD AND 4TH ORDERS

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Morphine content of opium-poppy sap. N. N. Vyroshtzov, Jr. and A. T. Troshchenko. *Compt. rend. acad. sci. U. R. S. S.* 2, 555-7(in German 558)(1935).—Sap analyzed not more than 2 hrs. after gathering showed 18.4 to 21.8% morphine. Controls allowed to stand until the next day showed the ordinary 12%. The diminution is attributed to oxidation by atm. O. Samples can be stabilized with KF for several months. G. M. R.

COMMON ELEMENTS

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

ca

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Production of phenol from chlorobenzene. N. N. Vorozhtsov, Jr. Org. Chem. Ind. (U. S. S. R.) 1, 823-5 (1936).—Various methods of production of PhOH from PhCl are discussed. In the production by the method of Hale and Britton (C. A. 32, 960) the loss of PhOH in soln., resulting from the decompn. of the 10% PhONa with dil. HCl or H_2SO_4, is eliminated by treating the phenolate with CO_2. After sepn. of the PhOH layer, the soln. contg. NaCl, Na_2CO_3 and some PhOH is treated with $Ca(OH)_2$ and filtered from the $CaCO_3$. After evapn. and filtration from NaCl, the soln., contg. NaOH and PhONa, is treated with NaOH to the desired concn. and then used in the hydrolysis of PhCl. The PhOH layer is treated with concd. H_2SO_4 to decompose any PhONa present, and then distd. Fifteen references. C. B.

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Cu

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Production of thiophenol and diphenyl sulfide from chlorobenzene. N. N. Vorozhtsov, Jr., and S. N. Mitsengendler. *Org. Chem. Ind.* (U. S. S. R.) 2, 457-61 (1936); cf. *C. A.* 28, 2340[9], 4073[1]. Chas. Blanc

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PROCESSES AND PROPERTIES INDEX

Ca

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The preparation of 8-hydroxyquinoline from 8-chloroquinoline. N. N. Vorozhtzov, Jr. and S. P. Mitzengendler. *J. Gen. Chem.* (U. S. S. R.) 6, 63 7(1936).

The direct prepn. of 8-hydroxyquinoline (I) from 8-chloroquinoline (II) by the action of dil. aq. NaOH is described. The 8-Cl is more reactive than either the 6-Cl or the Cl of PhCl. The app. was a rotary steel autoclave of 400-cc. capacity turning at 47 r. p. m. which was filled to the extent of 60%. The temp. range was 200-300°; the reaction time, 10 min. to 10 hrs., with NaOH concns. from 2-9%. Twice the theoretical amt. of NaOH was taken. The I formed was detd. bromometrically; the II which reacted, by weighing the NaCl produced. With 9% NaOH, 10 min., 90 atm. and 300°, 88% of II reacted and 64% of I was obtained. Reducing the temp. to 250° slowed up the reaction 12 times. At 225°, 10 hrs. are required for the conversion obtained at 300° in 10 min. With 2.2% NaOH, 2 hr. 40 atm. and 250°, 92.5% of II reacted and 87% of I was obtained. This time can be reduced to 20 min. with CuO catalyst but not with Cu.

Lewis W. Butz

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The preparation of 8-aminoquinoline from 8-chloroquinoline. N. N. Vorozhtsov, Jr., and S. P. Mitzengendler. *J. Gen. Chem.* (U. S. S. R.) 6, 681-4(1936); cf. *C. A.* 30, 4902.—8-Aminoquinoline (I), m. 64.5°, was obtained in 69.3% yield by heating 2.0 g. 8-chloroquinoline (II) with 20 mols. of 30% NH_3 and 0.20 g. CuCl catalyst in a rotary autoclave at 200° for 5 hrs. Increasing temp. decreased sharply the yields of I because of the decompn. of II. At 225° and 250° for 2.5 hrs. the decompn. of II increased to 76 and 83% and the yields of I decreased to 64 and 61.7%, resp. Cu_2O and Cu + $CuSO_4$ catalysts gave inferior results. Redistn. of II under atm. pressure resulted in partial decompn. of II with the formation of II.HCl, m. 169-71.5° (decompn.). No decompn. of II takes place by distn. at 10-mm. pressure. C. B.

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Esters of 3-phenylsalicylic acid. N. N. Vorozhtsov, Jr., and A. T. Troshchenko. Russ. 50,847, April 30, 1937. 3-Phenylsalicylic acid or its chloride is treated with the usual etherifying agents.

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2-Phenyl-1-phenol-4-sulfonic acid. N. N. Vorozhtsov, Jr., and A. T. Trushchenko. Russ. 50,857, April 30, 1937. o-PhC_6H_4OH is treated at ordinary or at increased temp. with concd. H_2SO_4.

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Acylated derivatives of 3-phenylsalicylic acid. N. N. Vorozhtsov, Jr., and A. T. Troshchenko. Russ. 50,840, April 30, 1937. 3-Phenylsalicylic acid is treated with acid anhydride or halide in the presence or absence of a catalyst.

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1ST AND 2ND CODES

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4-Nitro-2-phenyl- and 2-nitro-6-phenylphenol. N. N. Vorozhtsov, Jr., and A. T. Troshchenko. Russ. 51,218, June 30, 1937. In the nitration of 2-phenylphenol not more than 1.5 mols. HNO_3 is used. 2-Nitro-4-phenylphenol is steam-distd., and from the distn. residue 4-nitro-2-phenylphenol is sepd. in the usual manner.

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Nitrophenols, nitroanilines and their sulfo derivatives. N. N. Vorozhtsov, Jr. Russ. 51,507, Aug. 31, 1937. Polynitro compds. of Ph_2O or their sulfonic acids are heated with aq. NH_3 under pressure, and the reaction products are sepd. in the usual manner.

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Polynitro derivatives of the 4-sulfonic acid of diphenyl ether. N. N. Vorozhtsov, Jr. Russ. 51,628, Aug. 31, 1937. $PhOC_6H_4SO_3H$ is nitrated with H_2SO_4 and HNO_3 and the product sepd. in the usual way.

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Arylides of 2-hydroxydiphenyl-3 carboxylic acid N. N. Vorozhtsov, Jr., and A. T. Troshchenko. Russ. 51, [illegible], 1937. 2-Hydroxydiphenyl-3 carboxylic acid is heated with aromatic amines in the presence of condensing agents and a solvent. The arylide obtained is sepd. in the usual manner.

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2-Hydroxy-3-nitro-5-biphenylsulfonic acid N. N. Vorozhtsov, Jr. Russ. 51,872, Sept. 30, 1937. 2-Hydroxy-5-biphenylsulfonic acid is nitrated by known methods.

COMPOUND ELEMENTS

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Lacquer pigments. N. Y. Vorobitsov, Jr., V. V. Kozlov, A. V. Simanovskaya and A. T. Troshchenko. Russ. 52,804, March 31, 1938. Azo dyes obtained by coupling of *p*-hydroxybiphenyl with diazo compds. free of salt-forming groups are treated with a substrate such as $Al_2(SO_4)_3$ or $BaCl_2$.

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Derivatives of 2- and 4-hydroxybiphenyls. I. 2-hydroxy- and 4-hydroxybiphenyl-3-carboxylic acids and their derivatives. N. N. Vorozhtsov, Jr. and A. T. Troshchenko. *J. Gen. Chem.* (U. S. S. R.) 8, 424-9 (in English 430) (1938).—In the com. production of PhOH by decompn. of PhCl with NaOH at high pressure a considerable amt. of 2- (I) and 4-hydroxybiphenyl (II) is formed. The object of this investigation was to develop methods for the prepn. of various synthetic compds. from these by-products as a means for their com. utilization. I, m. 56.7° (petr. ether), and II, m. 163° (C_6H_6), were isolated by vacuum fractionation of the mixed phenols obtained from the distn. residue of PhOH by dissolving it in excess NaOH, driving off the neutral substances with steam and acidifying the soln. I forms an *Ac deriv.*, m. 64° (petr. ether), and *Bz deriv.*, m. 62° (alc.). II gives an *Ac deriv.*, m. 88-8.5°, and Bz deriv., m. 151-2° (alc.-toluene) (152°, Lachinov, *J. Russ. Phys.-Chem. Soc.* 5, 49(1873); 121°, Raiford and Colbert (*C. A.* 19, 1858). Autoclaving 30 g. II with 200 g. K_2CO_3 at 30 atm. CO_2 pressure and 200-50° for 5 hrs. gave 80% *4-hydroxybiphenyl-3-carboxylic acid* (III), m. 215-16° (C_6H_6). *Me ester*, m. 93-4°. III gave an *Ac deriv.*, $C_{13}H_{11}(OAc)CO_2H$, m. 151-2° (C_6H_6). Contrary to Ger. pat. 618,213 (*C. A.* 30, 300?), the arylamides of III ($PhNH_2$, 1-$C_{10}H_7NH_2$ and *m*-$O_2NC_6H_4NH_2$) show poor affinity for vegetable fibers. A yield of 84% *2-hydroxybiphenyl-3-carboxylic acid* (IV), m. 186-7° (C_6H_6), was obtained from 17 g. I and 51 g. K_2CO_3 at 160-80° and an optimum CO_2 pressure of 63 atm. (initial pressure 40 atm.) in 5 hrs. *Me ester*, m. 58-9° (MeOH); *Ph ester*, m. 90-2°. The arylamides obtained from IV were: *anilide*, m. 120-1°, *p-toluide*, m. 148-9°, and *p-chloroanilide*, 155-6°. IV and its arylamides when coupled with diazotized *p*-$O_2NC_6H_4NH_2$, benzidine and 1-$C_{10}H_7NH_2$ gave *azo dyes* and yellow to brown dyeings on fibers. Twelve references. II. **Nitrohydroxybiphenyls and aminohydroxybiphenyls and their derivatives. Azo dyes from hydroxybiphenyls.** *Ibid.* 431-7 (in English 437).—When 17 g. I in 100 ml. of 100% AcOH is treated slowly in the cold with 8 ml. HNO_3 (d. 1.39) the product is not *2-hydroxy-3,5-dinitrobiphenyl* (V) as assumed by Borsche (*Ann.* 312, 211(1900); *C. A.* 11, 2901), but a mixt. of 38.5% *2-hydroxy-3-nitrobiphenyl* (VI) and 37% *2-hydroxy-5-nitrobiphenyl* (VII). VII, m. 62° (alc.), is sepd. from VI, m. 123-4°, by steam distn. VI in Et_2O with CH_2N_2 gave *Me ether*, m. 72-3°. VI with concd. HNO_3 gave V, m. 203-4°, and this (5 g.) with 4 g. $NaNO_2$ and 50 g. of concd. H_2SO_4 on heating for 30 min. gave *2-hydroxy-3,5,2',4'-tetranitrobiphenyl*, yellow tablets, m. 182-3°. A mixt. of 50 g. VI in 200 ml. alc. with 160 g. $SnCl_2 \cdot 2H_2O$ in 400 ml. of concd. HCl was digested on a water bath for 12 hrs. and, after the addn. of 40 g. Sn, the heating was continued for 6 hrs. The reaction mixt., freed from the alc., was dild. to 2 l. and then satd. with H_2S and boiled. From the filtrate, on cooling, sepd. 41.5 g. (80.8% yield) *HCl salt of 2-hydroxy-3-aminobiphenyl* (VIII); the free base,

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colorless needles, m. 121-2.5°. *2-Methyl-7-phenylbenzoxazole*, m. 69-70°, (0.5 g. yield) was obtained when 3 g. VIII with 2 g. $AlCl_3$ in 5 ml. C_6H_6 was digested on a water bath for 3 hrs. VIII (3 g.) with 4 g. BzCl and 5 ml. C_6H_6 gave *2,7-diphenylbenzoxazole*, b_6 168-70°, m. 114-16°. *7-Phenyl-5-hydroxyquinoline*, m. 142-6°, was formed in 61% yield by refluxing for 3 hrs. a mixt. of 5 g. VIII, 7 g. VI, 15 g. of anhyd. glycerol and 8 g. of concd. H_2SO_4 and then driving off with steam unaltered VI.*HCl salt*, m. 208-9°. *4-Hydroxy-3-nitrobiphenyl* (IX) when reduced with Sn and HCl in dil. alc. gave a good yield of the amine (X). IX was obtained from II with HNO_3 (cf. Lachinov, *loc. cit.*). Refluxing for 16 hrs. 5 g. X.HCl, 2.8 g. IX, 15 g. glycerol, 7.5 g. of 100% AcOH and 5 g. H_2SO_4 and then steam-distg. the mixt. gave 95% *5-phenyl-8-hydroxyquinoline*, pale yellow needles, m. 91-2°. Diazotized VIII when coupled in alk. soln. with 2-$C_{10}H_7$ gave *3-hydroxybiphenyl-3-azo-2-naphthol*, red brown powder, m. 211-11.5°, and with resorcinol *2-hydroxy-3-azoresorcinol*. The following insol. azo dyes were prepd. by pouring a diazonium salt at 10-12° into an alk. II soln. *p-Nitrobenzeneazo-4-hydroxybiphenyl*, m. 174-5°. *1-Naphthylbenzeneazo-4-hydroxybiphenyl*, m. 121-3° (PhCl). *3,3'-Biphenylenediazobis[4-hydroxybiphenyl]*. *3,3'-(3,3'-Dimethylbiphenylenediazo)bis[4-hydroxybiphenyl]*. *(3,3'-Dimethoxybiphenylenediazo)bis[4-hydroxybiphenyl]*.

Chas. Blanc

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BC

A-3

Synthesis with α- and β-hydroxydiphenyls. II. Nitro- and amino-hydroxydiphenyls, their derivatives, and azo-dyes from hydroxydiphenyls. N. N. Vorozhtsov, jun., and A. T. Troshchenko (J. Gen. Chem. Russ., 1938, 8, 431—437).—α-C_6H_5·Ph·OH and HNO_3 in AcOH at 0° give a mixture of 2-nitro- (I), m.p. 62° (*Me ether*, m.p. 72—73°), and 5-nitro-2-hydroxydiphenyl. Further nitration of (I) yields successively 3 : 5-dinitro- and *tetranitro-2-hydroxydiphenyl*, m.p. 182—183°. (I) is reduced ($SnCl_2$ in EtOH) to *2-amino-2-hydroxydiphenyl* (II), m.p. 121—122·5°, which with AcCl in C_6H_6 (3 hr. at the b.p.) yields *6-phenyl-1-methylbenzoxazole*, m.p. 69—70°; with BzCl the product is *1 : 6-diphenylbenzoxazole*, m.p. 114—116°. (II), (I), and glycerol heated with H_2SO_4 yield *8-hydroxy-7-phenylquinoline*, m.p. 142—144° (*hydrochloride*, m.p. 208—209°; *Cu salt*). 3-Amino- and 3-nitro-*t*-hydroxydiphenyl give similarly *8-hydroxy-5-phenylquinoline*, m.p. 91—92° (*hydrochloride*, m.p. 212—226°; *Cu salt*). Diazotised (II) and β-$C_{10}H_7$·OH yield *2-hydroxy-3-β-hydroxynaphthalene-azodiphenyl*, m.p. 211—211·5°; an analogous *compound* with m-$C_6H_4(OH)_2$ is described. 4-Hydroxydiphenyl gives *azo-dyes* with diazotised p-NH_2·C_6H_4·NO_2, m.p. 174—175°, α-$C_{10}H_7$·NH_2, m.p. 121—123°, and with *tetrazotised* benzidine, 3 : 3'-dimethyl- and 3 : 3'-dimethoxy-benzidine.

R. T.

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BC

A1-3

Exchange of chlorine substituted in aromatic nuclei for the amino-group. III. Kinetics of catalytic reaction of chlorobenzene with aqueous ammonia. IV. Kinetics of reaction of p-chloronitrobenzene with aqueous ammonia. N. N. Vorozhtsov, Jun., and V. A. Koslov (J. Gen. Chem. Russ., 1933, 3, 1105—1119, 1330—1335).—II. The velocity of the reaction $PhCl + NH_3 \rightarrow NH_2Ph + HCl$, at 180—220°, ∝ concn. of PhCl and of catalysts (CuCl, CuBr, $CuCl_2$), but not to that of NH_3. Where increase in $[NH_3]$ accelerates the reaction this is due to presence of undissolved PhCl; the effect of increasing the $[NH_3]$ is thus to increase the effective [PhCl]. The reaction is retarded by NH_4Cl, to an extent ∝ $[NH_4Cl]$. The velocity coeff., $\log k = 6{\cdot}484 - 3606/T$ with CuCl, and $12{\cdot}52 - 6520/T$, with $CuCl_2$.

IV. The velocity of the reaction $p\text{-}C_6H_4Cl\cdot NO_2 + NH_3 \rightarrow p\text{-}NH_2\cdot C_6H_4NO_2 + HCl$ is ∝ concn. of substrates, and to the temp. ($\log k = 7{\cdot}24 - 4681/T$). In presence of $CuCl_2$ catalyst the velocity also ∝ $[CuCl_2]$. R. T.

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PROCESSES AND PROPERTIES INDEX

Cw

The replacement of aromatic chlorine by the amino group. IV. Kinetics of the interaction of *p*-nitrochlorobenzene with aqueous ammonia. N. N. Vorozhtsov, Jr., and V. A. Kobelev. *J. Gen. Chem.* (U. S. S. R.) 8, 1330-5(1933); cf. *C. A.* 33, 3709[1].—The reaction rate of $p\text{-}O_2NC_6H_4Cl$ (I) with aq. NH_4OH is directly proportional to the concn. of both constituents. The energy of activation of the reaction is 31,400 cal. The const. of the reaction velocity can be represented by the equation: $\log K_1 = 7.24 - (4661/T) \pm 0.01$. At 160° the velocity const. is 2.7×10^{-4} g./mol./l./min. In the presence of $CuCl_2$ the catalytic and noncatalytic reactions run parallel (cf. *C. A.* 29, 1787[1]). The velocity of the catalytic reaction is directly proportional to the concns. of I and the catalyst. At 160° this const. of velocity is 1.8×10^{-3} g./mol./l./min. Thus, the results accord with Groggins previous findings (*Chem. Met. Eng.* 36, 273 (1929)) and disagree with his later data (Groggins and Stirton, *C. A.* 30, 7977[1]). Chas. Blanc

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PROCESSES AND PROPERTIES INDEX

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Use of continuous processes in dye industry. N. N. Voroshtsov, Jr. *Org. Chem. Ind.* (U. S. S. R.) 6, 422-3 (1939).—The review of known processes for the continuous production of dye intermediates includes the production of phenols, naphthols and their derivs. from the corresponding chlorides and sulfonic acids by decompn. with aq. alkalies, obtaining of amines from PhCl and C_6H_5Cl with NH_4OH and reduction of nitro compds. with H in the presence of catalysts. Ten references. Chas. Blanc

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PROCESSES AND PROPERTIES INDEX

CA

Derivatives of 2- and 4-hydroxybiphenyls. III. 2-Hydroxybiphenyl-5-sulfonic acid and its derivatives. N. N. Yuzozhitsov, Jr., and A. T. Troshchenko. *J. Gen. Chem.* (U. S. S. R.) 9, 59-64(1939); cf. *C. A.* 32, 7907[5]. The sulfonation of *2-hydroxybiphenyl* (I) and the prepn. of the derivs. of *2-hydroxybiphenyl-5-sulfonic acid* (II) are studied for the first time. The reaction of 17 g. I with 15 g. of concd. H_2SO_4 on a water bath for 6 hrs. and with 30 g. acid at room temp. and 0-5° for several days produced identical II. The *Ca salt* (III) was isolated in 83.6% yield by pptn. with $CaCO_3$. Treating 4.3 g. III with 10 ml. Ac_2O at 150° for 5 hrs., expelling the excess of Ac_2O *in vacuo* and heating the residue with 5 g. PCl_5 on a water bath for 4 hrs. yielded 4.4 g. *2,5-AcO(SO$_2$Cl)C$_6$H$_3$Ph* (IV), m. 70-7° (C_6H_6). IV reacts with excess $PhNH_2$ in C_6H_6 by cleaving the Ac group and forming *HOC$_6$H$_3$(SO$_2$NH-Ph)Ph* (V), m. 146-7° (MeOH). The reaction at room temp. with a dropwise addn. of 2 mols. $PhNH_2$ proceeds without the Ac cleavage to form *AcOC$_6$H$_3$(SO$_2$NHPh)Ph* (VI), m. 141-2° (AcOEt). V and VI react with Ac_2O and NaOAc to yield *AcOC$_6$H$_3$(SO$_2$NAcPh)Ph*, m. 138-9° (AcOEt). Nitration of III gave the Ca salt of *2-hydroxy-3-nitrobiphenyl-5-sulfonic acid* (VII). It is obtained in nearly 100% yield when the II in the sulfonation mixt. (from 34 g. I), contg. 40 g. H_2SO_4, is heated in an oil bath at 115-25° for 6 hrs., the mixt. is dild. with 40 ml. H_2O and treated, with stirring, with 17 g. $NaNO_2$ in 50 ml. H_2O. The filtered ppt. is washed, dried and freed from 2-hydroxy-5-nitrobiphenyl by extn. with Et_2O. VII, treated with dil. HNO_3 on a water bath, gave 93% *2-hydroxy-3,5-dinitrobiphenyl*, m. 204°. VII (0.5 g.) on heating with 5 ml. of concd. HCl in a sealed tube at 160-80° for 10 hrs. is hydrolyzed to *2-hydroxy-3-nitrobiphenyl*, m. 62° (cf. *loc. cit.*). It can also be obtained by refluxing VII with 50% H_2SO_4. Treating 0.5 g. of the Na salt (VIII) of VII in 20 ml. of cold H_2O with 4 ml. of 5.6% $KBrO_3$ and 4 ml. of concd. HBr yielded 0.33 g. *2-hydroxy-3-nitro-5-bromobiphenyl*, m. 113-15° (petr. ether). The corresponding Cl *deriv.* was prepd. (1) from VIII in H_2O with gaseous Cl, and (2) by nitrating 2-hydroxy-5-chlorobiphenyl (cf. U. S. 1,921,727, *C. A.* 27, 5186). Recrystd. from a mixt. of petr. ether and C_6H_6, it m. 127.5-8°. Reduction of VIII with Sn in 50% HCl yielded 70.5% *2-hydroxy-3-aminobiphenyl-5-sulfonic acid*, a stable compd., fairly sol. in hot water. In H_2O it reacts with $FeCl_3$ to give a red-brown soln., forming rapidly a dark ppt., and with HNO_2 a diazo compd., capable of coupling with the common azo components. It gives an azo compd. with resorcinol, dyeing wool yellow-brown, which on chroming becomes brown-red, and with 2-naphthol a dark violet dye, giving reddish dyeing on wool, which on chroming becomes dark violet. C. B.

METALLURGICAL LITERATURE CLASSIFICATION

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PROCESSES AND PROPERTIES INDEX

The replacement of aromatic chlorine by the amino group. V. Kinetics of the interaction of o-nitrochlorobenzene with aqueous ammonia. N. N. Yorozhtsov, Jr., and V. A. Kobelev. *J. Gen. Chem.* (U. S. S. R.) 9, 1043-6(1939); *cf. C. A.* 33, 4114[8].—The rate of reaction of

o-$O_2NC_6H_4Cl$ with aq. NH_4OH is directly proportional to the concn. of both reactants. The energy of activation of the reaction is 20,500 cal. The const. of the reaction velocity can be represented by the equation: $\log K = 7.20 - (4452/T) \pm 0.01$. At 100° the velocity const. is 7.1×10^{-4} g. mol./l./min. In the presence of $CuCl_2.2H_2O$ the catalytic and noncatalytic reactions run parallel. The velocity of catalytic reaction is considerably greater than that of noncatalytic reaction. At 100° this const. of velocity is 0.1 g. mol./l./min. A. A. Podgorny

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PROCESSES AND PROPERTIES INDEX

Cu

Comparative reactivities of certain chloro- and bromonitrobenzenes. N. N. Vorozhtsov, Jr., and V. A. Kobelev. *J. Gen. Chem.* (U. S. S. R.) 9, 1047-8(1939).—The reaction of *o*- and *m*-$ClC_6H_4NO_2$ was carried out by the Sprung method (cf. *C. A.* 24, 2430). The progress of the reaction was followed by the change of sulfite titer and by the formation of chloride ion. Conclusions: No reaction between the reactants was observed at all. The disappearance of sulfite was caused by its autoxidation, the velocity of which varied and depended on a series of undetd. causes. No chloride ion formation was observed in the expts., and practically all chlorobenzenes were recovered from the reaction mixt. Thus, the data obtained by Sprung are not correct. A. A. Podgorny

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CA

The replacement of aromatic chlorine by the amino group. VI. Kinetics of the interaction of m-nitrochlorobenzene with aqueous ammonia. N. N. Vorozhtsov, Jr., and V. A. Kobelev. *J. Gen. Chem.* (U. S. S. R.) 9, 1465-7 (1939); cf. *C. A.* 33, 8580.—Unlike o- and p-$O_2NC_6H_4Cl$ the m-isomer (I) does not react at all with aq. NH_3 at 200° and but slightly (3%) at 230°. In the presence of $CuCl_2.2H_2O$ the rate of ammonolysis is directly proportional to the concn. of I and the catalyst and is independent of the NH_4OH concn. The energy of activation of the reaction is 13,700. The const. of the reaction velocity can be represented by the equation: $\log K = 5.387 - (2633/T)$. VII. Kinetics of the interaction of β-chloroanthraquinone with aqueous ammonia. *Ibid.* 1518-16.—The rate of reaction of β-chloroanthraquinone with aq. NH_3 is directly proportional to the concn. of both reactants. The energy of activation of the reaction is 20,500. The const. of the reaction velocity is: $\log K = 6.26 - (4467/T)$. At 210° the velocity const. is 10.37×10^{-4}. The reaction is best effected in the presence of the $Cu(NO_3)_2$ catalyst (cf. Vorozhtsov and Nikitin, *C. A.* 32, 539[9]). VIII. Kinetics of the interaction of p-chloroaniline, 1-chloronaphthalene and sodium 1,4-chloronaphthalenesul-

fonate with aqueous ammonia in the presence of copper chloride. *Ibid.* 1509-70.—The rate of reaction of p-$ClC_6H_4NH_2$ (I), 1-$C_{10}H_7Cl$ (II) and 1,4-$ClC_{10}H_6SO_3Na$ (III) with aq. NH_3 in the presence of $CuCl_2$ is directly proportional to the concn. of the org. chlorides and the catalyst and is independent of the NH_4OH concn. The cumulative evidence of this investigation series shows that in the catalytic interaction with NH_4OH the org. Cl derivs. form the intermediate complex $RCl.Cu(NH_3)_4$ which then reacts with excess NH_4OH to give aromatic amines. The energy of activation of the reaction of I, II and III, resp., are: 12,500, 12,000 and 12,200. The corresponding consts. of the reaction velocity can be represented by the equations: $\log K = 4.05 - (2732/T)$; $\log K = 3.98 - (2632/T)$, and $\log K = 4.314 - (2677/T) = 0.005$. The soly. of II in NH_4OH is greater than in H_2O and increases with NH_4OH concn.

Chas. Blanc

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VOROZHTSOV, Jr., N.N.; KOBELEV, V. A.

"The Change of Aromatically-Combined Chlorine into an Amino Grouping" Part VII. "The Kinetics of the Reaction B-Chloroanthraquinone with Aqueous Solutions of Ammonia", Zhur. Obshch. Khim., 9, No. 16, 1939. State Institute of High Pressures, Leningrad. Rec'd 20 Feb 1939.

Report U-1614, 3 Jan 1952.

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VOROZHTSOV, Jr., N.N.; KOBELEV, V.A.

"The Change of Aromatically-Combined Chlorine into an Amino Grouping" Part VIII. "The Kinetics of the Reaction of p-Chloraniline, Chloronaphthalin, and the Sodium Salt of 1,4-Chlorosulfonic Acid of Naphthalin with Aqueous Solutions of Ammonia in the Presence of Copper Chloride", Zhur. Obshch. Khim., 9, No. 17, 1939. State Institute of High Pressures, Leningrad. Received 28 Feb 1939.

Report U-1614, 3 Jan 1952.

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CA

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Nitro derivatives of diphenyl-ether-4-sulfonic acids. N. N. Vorozhtsov, Jr. *J. Gen. Chem.* (U. S. S. R.) 10, 835-41(1940); cf. Russ. pat. 51,828, *C. A.* 33, 6880[1].—Nitrosulfonic acids of Ph_2O (I), which is easily accessible as a by-product in the manuf. of phenol from PhCl, have been prepd. and investigated. I (21 g.), warmed with 10 ml. concd. H_2SO_4, cooled, and treated with 13 ml. HNO_3 (d. 1.4) and 20 ml. concd. H_2SO_4 within 30 min., first at 35-40° while stirring and cooling and then for 15 min. at 70-80°, yields *3-nitro-4-(4'-nitrophenoxy)benzenesulfonic acid* (II), isolated as the *Na salt.3H_2O* (III) (yield 7.5 g.); *Ba salt; chloride* (IV), m. 134-6.5°, from III on heating with PCl_5; *amide*, m. 188-90°. When 21 g. I is heated with 14 ml. H_2SO_4, the reaction mixt. at 35-45° is treated with 36 ml. H_2SO_4 and 36 ml. HNO_3 (d. 1.4), and the whole then warmed for 2.5 hrs. to 90°, there is obtained *3-nitro-(2',4'-dinitrophenoxy)-benzenesulfonic acid* (V), isolated as the *Na salt.H_2O* (VI) (yield 21 g.); *chloride* (VII), m. 157-9°, from VI on heating with PCl_5. VI (1 g.) in 4 ml. concd. H_2SO_4 is treated with 0.3 ml. HNO_3 (d. 1.4) on the water bath to yield *bis(2,4-dinitrophenyl) ether* (VIII), m. 104-4°. III on boiling with NaOH soln. followed by acidification yields 6-nitrophenol (IX) and 2-nitro-1-phenol-4-sulfonic acid (X). VI yields 2,4-dinitrophenol (XI) and X when treated similarly. Cleavage of III by means of concd. NH_3 soln. gives IX and 3-nitrosulfanilic acid, whereas VI under analogous conditions gives 2,4-dinitroaniline (XII) and X. Cleavage of IV and VII with concd. ammonia yields 4-amino-3-nitrobenzenesulfonamide and IX, and XII and *2-nitro-1-phenol-4-sulfonamide*, m. 283-6.5°, resp., the latter compd. being identical with the amide obtained directly from X. Cleavage of VIII gives XI and XII by means of concd. NH_3, and 2,4-dinitrodiphenylamine and XI by means of aniline.

Gertrude Berend

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

ca

10

Derivatives of isocoumarin. I. Synthesis of isocoumarin-3-carboxylic acid. N. N. Vorozhtsov, Jr., and L. N. Bogusevich. *J. Gen. Chem.* (U. S. S. R.) 10, 2014-16(1940).—The di-Me ester of isocoumarin-3,4-dicarboxylic acid (I) was obtained by the condensation of di-Me homophthalate with $(CO_2Me)_2$ (in ether in the presence of metallic Na). Isocoumarin-3-carboxylic acid (II) was obtained by refluxing 2 g. of I for 2 hrs. with 10 ml. concd. HCl. Thin colorless needles of II, m. 236°, pptd. on cooling the soln. It melted at the same temp. when mixed with II prepd. from β-naphthoquinone according to Bamberger and Kitschelt (*Ber.* 25, 800, 1138 (1892)). The Me ester of II, m. 174°, was obtained when II was methylated with CH_2N_2. II was also obtained on condensation of di-Et homophthalate with $(CO_2Et)_2$, after heating the condensation product with HCl.

James J. Lichtin

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Ca

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Catalytic oxidation of phenanthrene. I. Influence of various factors on the process of catalytic oxidation of phenanthrene. N. N. Vorozhtsov and D. A. Gerevich. (Kafedra Tekhnol. Promezhutochnykh Produktov i Krasitelei Moskov. Khim. Tekhnol. Inst. im. D. I. Mendeleeva). *J. Applied Chem.* (U.S.S.R.) 18, 3-9(1945).—The vapor-phase oxidation of phenanthrene was studied with the following catalysts: Cu oxide, Cr oxides, Cu vanadate, mixts. of V_2O_5 with MoO_3; pure V_2O_5 and V_2O_5 with addends of compds. of Na, K, Cs, Ca, Co, and Mn. 9,10-Phenanthrenequinone and phthalic anhydride were formed only with catalysts contg. V_2O_5; other catalysts led, to a greater or lesser degree, to complete oxidation to H_2O and CO_2. The highest conversions were obtained with the most rigorously purified V_2O_5, when the yield of acidic products, calcd. as phthalic anhydride, reached 74.8%; V_2O_5 from com. NH_4 vanadate and promoted by salts of metals of the 1st group gave slightly lower yields (73-4%); addn. of MoO_3 somewhat raises the yield of phenanthrenequinone (7% being approx. the highest yield) while addn. of Cu salts leads to deep oxidation. Optimum yields are attained at 400°, while lower temps. do not favor the yields of phenanthrenequinone, which is always subordinated by phthalic anhydride formation. Increase of space velocity increases the unit volume efficiency of the catalyst but leads to poorer quality of product. **II. Mechanism of action of vanadium pentoxide.** *Ibid.* 10-14.—Photomicrographic examn. of used catalyst shows appreciable changes, apparently by multifold oxidation-reduction. Increase of the disperse state of the top layers of the catalyst improves its efficiency. It is proposed that the activation of the catalyst surface depends upon its improved dispersed character by oxidation-reduction agents. Fusion of the catalyst which leads to macrocryst. state drops the activity considerably. G. M. Kosolapoff

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

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CA

18

Activating vanadium pentoxide oxidation catalysts. N. N. Vereshtsov and D. A. Gurevich. U.S.S.R. 65,703, Jan. 1, 1946. The activity of V_2O_5 catalysts increases with the degree of dispersion. To increase the degree of dispersion the catalyst is treated first with a reducing agent, e.g., H, and then with O at 400-50°. M. Hosch

COMMON ELEMENTS

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ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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VOROZHTSOV.N.N.

Manufacture of intermediate products in German plants. Khim.prom. no.6:176-181 Je'47. (MLRA 8:12)
(Germany, West--Chemical industries)

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10

The reaction of aromatic sulfonic acids with phenols. N. N. Vorozhtsov and A. B. Kuchkarov (Mendeleev Inst. Chem. Technol., Moscow). *J. Gen. Chem.* (U.S.S.R.) 19, No. 10, 1413-20(1949)(English translation).—See C.A. 44, 1022e. E. J. C.

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CA

10

Reaction of aromatic sulfonic acids and phenols. N. N. Vorozhtsov and A. B. Kuchkarov (Lab. Krasitelei, Moskov. Khim.-Tekhnol. Inst. im. Mendeleeva). *Zhur. Obshchei Khim.* (J. Gen. Chem.) **10**, 1943-50(1940). PhOH (9.4 g.) and 7.9 g. $PhSO_3H$ after 12 hrs. at 290-40° gave 37% *p-hydroxydiphenyl sulfone*, m. 135.6° (from C_6H_6); *di-Br deriv.*, m. 231-2° (from EtOH), prepd. by bromination in AcOH at 18°; *mononitro deriv.*, m. 131-2° (from EtOH), obtained with mixed acid in AcOH or with fuming HNO_3 in AcOH; *MeO deriv.*, m. 90° (from EtOH), by heating in alkali with Me_2SO_4; *Ac deriv.*, m. 95° (from EtOH), by Ac_2O treatment. Similarly, *p*-cresol (10.8 g.) and 7.9 g. $PhSO_3H$ gave 20%, *2-hydroxy-5-methyldiphenyl sulfone*, m. 137-8° (from EtOH), while *o*-cresol gave 11.5%, *4-hydroxy-5-methyldiphenyl sulfone*, m. 290-1° (from EtOH); *MeO deriv.*, m. 101-2° (from EtOH); *Ac deriv.*, m. 98° (from EtOH); *nitro deriv.*, m. 140° (from EtOH); *Br deriv.*, m. 230° (from EtOH). Similarly, *m*-cresol gave 27%, *4-hydroxy-6-methyldiphenyl sulfone*, b. 280-5°, m. 120° (from EtOH); *di-Br deriv.*, m. 175°; *dinitro deriv.*, m. 172-3° (by the action of concd. HNO_3 in AcOH); *Ac deriv.*, m. 137-8°. Similar reaction of 18.8 g. PhOH and 19.2 g. *p*-$ClC_6H_4SO_3H$ gave 15% 4-hydroxy-4'-chlorodiphenyl sulfone, m. 153-6° (from EtOH), and *o*-cresol gave 33% *4-hydroxy-5-methyl-4'-chlorodiphenyl sulfone*, m. 220-30° (*nitro deriv.*, m. 157-8°), giving a red dye with diazotized *p*-nitroaniline. Treatment of 5,1-$Me(HO)C_6H_3SO_2Ph$ (2.48 g.) in 25 ml. 0.5 *N* NaOH with 0.72 g. $NaNO_2$ at 0°, followed by *N* H_2SO_4, gave no reaction, but a similar reaction with $(p\text{-}HOC_6H_4)_2SO_2$ succeeded only with a 4-fold reagent excess, yielding a di-NO_2 deriv., m. 232-3°, instead of the expected nitroso deriv. G. M. Kosolapoff

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CA

Addition of aniline to double bonds of olefins in the presence of heterogeneous catalysts. N. N. Yurgshtsev and I. I. Ioffe. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 1650-60(1951). Heating 20 g. $Me_2CHCH:CH_2$ and 55 g. $PhNH_2$ 13.5 hrs. in an autoclave at 250-60° and 25-9 atm. with 25 g. activated kaolin catalyst (250 g. sieved kaolin boiled 12 hrs. with 1.51. 25% H_2SO_4, washed by decantation until neutral, dried, and pressed into tablets) placed in the vapor phase of the mixt. gave after the usual treatment with HCl and NaOH 8.8 g. *p-tert-amylaniline* (insol. sulfate; *N*-Ac deriv., m. 137°) and 0.82 g. mixed secondary amines which with *p*-$MeC_6H_4SO_2Cl$ gave *2-methyl-3-(phenyl-p-tolylsulfamido)butane*, m. 83°, while the unreacted residue with HNO_2 gave a test for nitroso derivs., possibly because of the presence of *2-aniline-2-methylbutane*. The results may be explained by isomerization of the olefin to $Me_2C:CHMe$. G. M. K.

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VOROZHTSOV, N. N. Chechoslovakia CA: 47:12823

Zaklady synthesy polotovaru a barviv. 3rd ed.

Prague: Tech.-vedecke vydavatelstvi. 1952. 302 pp. Kcs. 250.

Reviewed in Chem. Listy 47, 946 (1953).

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4

Preparation of acetyl-p-benzoquinone and some of its derivatives. N. N. Vorozhtsov and V. P. Mamaev. D. I. [illegible] Chem.-Technol. Inst. Moscow [illegible]

[illegible] solid with fresh [illegible] red-orange 2-acetyl-1,4-benzoquinone (I), [illegible]

[illegible] the above product is a [illegible] form, in [illegible] Hydrolysis of this [illegible] at reflux gave a product with 2 HO [illegible] probably [illegible] gave a green [illegible]

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VOROZHTSOV, N. M.

[illegible] Mendeleev Chem. [illegible] Khim. [illegible] Oxidation of o-[illegible] with [illegible] H_2SO_4 gave toluquinone (I), m. [illegible] Refluxing [illegible] I with [illegible] ml. C_6H_6 and 6.7 g. [illegible] gave a dark ppt. (I [illegible]) and a red soln. The soln. on evapn. gave yellow 5-chloro-2-methyl-1,4-benzoquinone (II), m. 104.3–5.5° (from dil. EtOH). This (1 g.) in H_2O treated with SO_2 and the satd. soln. allowed to stand overnight, then extd. with Et_2O and the ext. evapd. gave 5-chloro-2-methylhydroquinone, m. 178–4°. The same was found to be the principal constituent of the dark ppt. formed in the original reaction (above) on crystn. from petr. ether; crystn. from H_2O gave red needles, m. 125–6.5°, which is apparently a quinhydrone-type complex of the quinone and the hydroquinone; since treatment of this with SO_2 gave the hydroquinone deriv., m. 172–4°. When 4.6 g. I in C_6H_6 was refluxed 5 hrs. with 5.3 g. SO_2Cl_2 and 0.01 g. Bz_2O_2, there was obtained 1.65 g. quinhydrone deriv. and 3 g. II. Thus the Bz_2O_2 added merely changed the rate of the reaction but not its course. G. M. Kosola

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VOROZHTSOV, N.N.

USSR/Scientists - Chemistry

Card 1/1 : Pub. 151 - 37/37

Authors : Rodionov, V. M.; Vorozhtsov, N. N.; Smirnova, A. F.; Shchetinina, L. A.; Shestov, A. P.; Korolev, A. I.; Lukashevich, V. O.; and Ufimtsev, V. N.

Title : In memory of Evgeniy Alekseevich Ivanov

Periodical : Zhur. ob. khim. 24/3, 579-580, Mar 1954

Abstract : Eulogy is presented honoring the passing of E. A. Ivanov, chief of the Central Laboratory of the Dorogomilov-Frunze Chemical Plant, scientist in the field of organic semi-products and dyes, recipient of Stalin premium. Illustration.

Institution: .....

Submitted : .....

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

VOROZHTSOV, N.N.

USSR/Chemistry

Card 1/1

Authors : Vorozhtsov, N. N. Jun.; and Beskin, A. M.

Title : Catalytic conversions of halogen derivatives of the aromatic series. Part 1.- Isomerization of monochloro-naphthalines.

Periodical : Zhur. Ob. Khim. 24, Ed. 4, 657 - 664, April 1954

Abstract : Experiments showed that 1-chloronaphthalin, when passing its vapors over an aluminosilicate catalyst or over aluminum oxide in the stream of hydrogen chloride, smoothly isomerizes into 2-chloronaphtalin. The isomerization reaction is reversible. In these conditions the 2-chloronaphthalin converts into 1-chloronaphthalin. Equilibrium at 350° is attained by a mixture of isomers containing about 55% of 2-chloronaphthalin. Eleven references; 5 USSR since 1875; 3 German since 1934; 3 French since 1886. Tables, graph.

Institution : The K. E. Voroshilov Scientific-Research Institute of Organic Semi-products and Dyes.

Submitted : November 19, 1953

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VOROZHTSOV, N.N.

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Catalytic transformations of halogen derivatives in the aromatic series. I. Isomerization of the monochloronaphthalenes. Vorozhtsov, N. N. and A. M. Reskin. Zhurnal Obshchei Khimii, 24, 1954.

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Voronezh Tsov, P.N.

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"Catalytic transformations of halogen derivatives of the [illegible] of isomerization of [illegible] N. Voronezh [illegible] 1959 [illegible]

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VORDZHITSOV, N.N.

Catalytic transformations of halogen derivatives of the [illegible]

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VOROZHTSOV, N.N.

USSR/Chemistry - Catalytic conversion

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Authors : Vorozhtsov, N. N., Junior, and Przhiyalgovskaya, N. M.

Title : Catalytic conversions of halogen derivatives of the aromatic series. Part 2. Interchange reaction during the reaction of halogeno-naphthalenes with hydrogen halides

Periodical : Zhur. ob. khim. 24/10, 1787-1795, Oct 1954

Abstract : The interchange reaction in halogen derivatives of the aromatic series was investigated at 350° in the presence of Al_2O_3. The presence of the catalyst (Al_2O_3) was found to be a necessary condition for the interchange reaction as well as isomerization. The interchange reaction of chloronaphthalene and bromonaphthalene results in the formation of naphthalene and its dihalogeno-derivatives. It was established that the latter products form as result of reversibility of the naphthalene bromination reaction. The possibility of converting bromobenzene into chlorobenzene under the effect of hydrogen chloride is explained. Twenty-three references: 14-German; 3-USA and 6-USSR (1905-1954). Tables; graph.

Institution : The D. I. Mendeleyev Chemical-Technical Institute, Moscow

Submitted : April 9, 1954

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VOROZHTSOV, Nikolay Nikolayevich, 1881-1941; VOROZHTSOV,N.N.(Jr.), redaktor; SHVETSOV,Yu.B., redaktor; LUR'YE,M.S., tekhnicheskiy redaktor; POGUDKIN,P.V., tekhnicheskiy redaktor

[Fundamentals of the synthesis of intermediate products and dyes] Osnovy sinteza promezhutochnykh produktov i krasitelei. 4-e izd. Moskva, Gos.nauchno-tekhn. izd-vo khimicheskoi lit-ry, 1955. 839 p. (MIRA 9:3)

(Chemistry, Organic--Synthesis) (Synthetic products) (Dyes and dyeing--Chemistry)

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"APPROVED FOR RELEASE: 03/14/2001 CIA-RDP86-00513R001861020003-0

VOROZHTSOV, N.N., professor.

Conference of chemists in Salzburg. Khim. nauka i prom. 1 no.6:716-717 '56. (MLRA 10:3)

(Salzburg--Chemistry--Congresses)

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