"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A.

How to prevent violations. Fin. SSSR 23 no.10:68-71 O '62. (MIRA 15:10)

1. Zamestitel' glavnogo kontrolera-revizora Kontral'no-revizionnogo upravleniya Ministerstva finansov RSFSR po Leningradu.
(Leningrad--Auditing and inspection)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A.

New stores in Volgograd Sov torg 35 no.1:55-57 Ja '62. (MIRA 15:1)

(Volgograd--Retail trade)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

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APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

FIL'SENKO, [illegible] ROV, A

[illegible] "[illegible]" [illegible]
Zemleme[illegible]

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

VELKOV, V., prof.; PETROV, A. [translator]; NANCHEVA, T. [translator]; GERGOVA, Marina, red.; IVANOV, Aleksandr, tekhn. red.

[Pruning fruit trees] Obrezka plodovykh derev'ev. 2. stereotipnoe izd. russkogo perevoda. Sofiia, Gos. izd-vo sel'khoz. lit-ry, 1961. 222 p. (MIRA 15:4)

1. Zaveduyushchiy Kafedroy plodovodstva Vysshego sel'skokhozyaystvennogo instituta im. V.Kolarova v g. Plovdiv (for Velkov).
(Pruning) (Fruit trees)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A., polkovnik

Petty officers should follow the example of the best of them.
Komm.Vooruzh.Sil 1 no.5:70-74 D '60. (MIRA 14:8)
(Russia--Navy--Petty officers)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

LOGINOV, V.; VOYNOV, A.; BARANOVA, V.; PETROV, A.

To all young engineers and technicians, agricultural specialists, students of institutions of higher learning and technical schools. NTO 2 no.10:5-6 O '60. (MIRA 13:10)

1. Sekretar' partiynogo byuro Yaroslavskogo zavoda toplivnoy apparatury (for Loginov). 2. Predsedatel' zavkoma profsoyuza Yaroslavskogo zavoda toplivnoy apparatury (for Voynov). 3. Sekretar' Vsesoyuznogo Leninskogo kommunisticheskogo soyuza molodezhi Yaroslavskogo zavoda toplivnoy apparatury (for Baranova). 4. Predsedatel' soveta nauchno-tekhnicheskogo obshchestva Yaroslavskogo zavoda toplivnoy apparatury (for Petrov).
(Technological innovations)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A.

Management decides. Grazhd.av 17 no.9:6-8 S '60. (MIRA 13:9)

1. Zamestitel' komandira podrazdeleniya, chlen byuro partiynoy organizatsii Severnogo territorial'nogo upravleniya Grazhdanskogo vozdushnogo flota.

(Aeronautics, Commercial)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A.

Preparations for the winter season have been completed on July 15th.
Zhil.-kom. khoz. 10 no.10:15-17 '60. (MIRA 13:10)
(Moscow--Apartment houses--Maintenance and repair)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A.

[illegible] the method of the [illegible] field analysis [illegible]
herbaceous plants. Doklady [illegible] 17 no. [illegible] [illegible]

1. Institute of Fruit Culture, Plovdiv. Submitted [illegible]

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

DOROKHOV, S.M.; LYAYMAN, E.M.; KASPIN, B.A.; SOLOV'YEV, T.T.; MARTYSHEV, F.G., prof., nauchnyy red.; PETROV, A.A., red.; UDALOV, A.G., tekhn.red.

[Fish culture on farms] Sel'skokhozialstvennoe rybovodstvo. Moskva, Izd-vo M-va sel'khoz.SSSR, 1959. 198 p. (MIRA 13:6)
(Fish culture)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

SOMOV, G.P., SHAPIRO, M.I., PETROV, A.A.

Studies on an island focus of North Asiatic tick-born typhus.
Zhur.mikrobiol.epid. i immun. 29 no.5:94-99 My '58 (MIRA 11:6)
(TYPHUS, epidemiology
in Russia, focus of North Asia tick-borne infect.
on island (Rus))

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

KALININ, Mikhail Semenovich; PETROV, A.A., red.; BARBASH, F.L., tekhn.red.

[Corn varieties and hybrids] Sorta i gibridy kukuruzy. Moskva, Izd-vo M-va sel'. khoz. SSSR, 1958. 124 p. (MIRA 12:8)
(Corn (Maize)--Varieties)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

KISIN, V.M., inzhener; PETROV, A.A.

Furniture made of standardized blanks and parts joined without tenons. Der.prom 5 no.12:3-5 D '56. (MLRA 10:1)
(Furniture industry) (Joinery)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A.A., inzh.

Sectional chair with plywood sides. Der.prom.6 no.12:19-20 D '57 (MIRA 10:12)

(Chairs)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

KUPIN, B.S.; PETROV, A.A.

Course of the hydration of disubstituted acetylenes. Part II: Hydration of isopropyl-, tert-butyl-, and isopropenylacetylenes. Zhur.ob.khim. 33 no.12:3860-3863 D '63. (MIRA 17:3)

1. Leningradskiy tekhnologicheskiy institut imeni Lensoveta.

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

BLEKHMAN, Aron Borisovich, inzh.; PETROV, A.A., red.; LEBEDEVA, I.D., red. izd-va; VDOVINA, V.M., tekhn. red.

[Matching of planed veneer for furniture facing] Podbor stroganoi fanery i shpona dlia oblitsovki mebeli. Moskva, Goslesbumizdat, 1961. 85 p. (MIRA 15:3)
(Veneer and veneering) (Furniture)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A.

Chemical Abst.
Vol. 48 No. 8
Apr. 25, 1954
Biological Chemistry

(5)

Chronic and acute intoxication by manganese. M. Iotsov, P. Ovcharova, R. Koinov, and A. Petrov. *Annuaire Acad. Med. "Valko Tchervenkov"* 30, 437–41(1950–51)(French summary).—Lethal intoxication of rabbits with $MnCl_2$ did not cause any pathol. changes in the blood-cerebral barrier and did not influence the passage of trypan blue or NaI into the cephalo-rhachidian liquid or to the brain tissues. Chronic intoxication with a total of 180 mg. $MnCl_2$/kg. in 2% soln. during 5 months gave similar results. Lethal and toxic doses depend both on the quantity of $MnCl_2$ and the concn. of the solns. used. G. Meguerian

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

SERGIYENKO, S.R.; GARBALINSKIY, V.A.; PETROVA, A.A.; CHIROVA, Ye.V.; MURADOVA, G.A.

Composition and properties of hydrocarbons from condensates of the Islim deposit. Izv. AN Turk. SSR. Ser. fiz.-tekh., khim. i geol. nauk no.1:37-47 '65. (MIRA 18:7)

1. Institut khimii AN Turkmenskoy SSR.

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A. A.

Kolmogorov, A. N., Petrov, A. A., and Smirnov, Yu. M. A formula of Gauss in the theory of the method of least squares. Izvestiya Akad. Nauk SSSR. Ser. Mat. 11, 561-566 (1947). (Russian)

In articles 39-40 of Gauss's Theoria Combinationis Observationum Erroribus Minimis Obnoxiae there occurs the inequality $\rho\rho/\pi \leqq \sum(a\alpha+b\beta+c\gamma+\cdots)^2<\pi$. Gauss failed to notice that this inequality can be sharpened. The purpose of the paper is to show that $\rho\rho/\pi \leqq \sum(a\alpha+b\beta+c\gamma+\cdots)^2 \leqq \rho$ and that this latter inequality cannot be improved.

W. E. Milne (Corvallis, Ore.).

Source: Mathematical Reviews, Vol 9 No. 7

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A.A., inzhener.

Tolerances for non-limited dimensions. Vest.mash. 33 no.10:100-101 O '53. (MLRA 6:10)
(Tolerance (Engineering))

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A.A.

Vertification of statistical hypotheses on the type of a distribution based on small samples [with summary in English]. Teor. veroiat. i ee prim. 1 no.2:248-271 '56. (MLRA 10:2)

(Distribution (Probability theory))

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PHASE 1 BOOK EXPLOITATION SOV/3104

Moscow. Inzhenerno-fizicheskiy institut. Kafedra matematiki

Sbornik nauchnykh rabot (Collection of Scientific Works) [Moscow?] Atomizdat, 1958. 194 p. 1,000 copies printed.

Tech. Ed. R.A. Negrimovskaya.

PURPOSE: This book is intended for applied mathematicians and theoretical physicists.

COVERAGE: This book contains a collection of articles by leading Soviet mathematicians and physicists on topics in atomic physics. The Thomas-Fermi generalized model of the atom, distributions, compression of an isothermic gas, boundary value problems, etc. are discussed. The book was published under the auspices of the Department of Mathematics of the Moscow Engineering-Physics Institute. No personalities are mentioned. References accompany individual articles.

Card 1/3

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"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

SOV/3104

Collection of Scientific Works

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APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

L 9438-66 EWT(d)/EWT(m)/EWP(f)/T-2/EWA(c)

ACC NR: AP5028524 SOURCE CODE: UR/0286/65/000/020/0112/0112

INVENTOR: Alekseyev, O. N.; Petrov, A. A.

31
B

TITLE: A device to scavenge a two-cycle diesel with a gas turbine supercharger. Class 46, No. 175790 [announced by the Bryansk Machine Building Plant (Bryanskiy mashinostroitel'nyy zavod)]

44,55

SOURCE: Byulleten' izobreteniy i tovarnykh znakov, no. 20, 1965, 112

TOPIC TAGS: diesel engine, supercharger

ABSTRACT: An Author Certificate has been issued for a device to scavenge a two-cycle diesel using gas-turbine supercharging and the compression of the scavenging air in the cavity below the pistons (see Fig. 1). It has a supplementary chamber

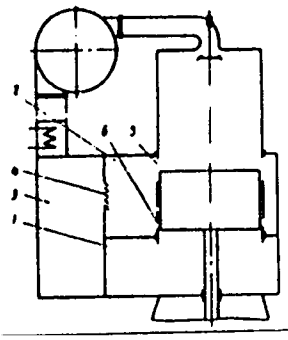

Fig. 1. Device to scavenge a two-cycle diesel with a gas-turbine supercharger

1 - Below-piston cavity; 2 - supplementary chamber; 3 - receiver; 4 - one-way valve; 5 - scavenging port; 6 - second row of scavenging ports.

Card 1/2 UDC: 621.436

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"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

L 9438-66

ACC NR: AP5028524 0

connected with a receiver by one-way valves, and with the working cylinder by scavenging ports. To increase the engine's efficiency, a supplementary chamber is connected with the cavity below the pistons by a second row of ports controlled indirectly either by the lower port of the working piston or by a valve driven by the piston rod. Orig. art. has: 1 figure. [WH]

SUB CODE: 21/ SUBM DATE: 14Aug64/ ATD PRESS: 4156

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Card 2/2

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

RADCHENKO, S.I., PETROV, A.A.

Alkylthienyles and their analogs. Part 2. Addition of sulfenyl chlorides to vinylacetylene. Zhur. obshch. khim. 1 no.1 47-51 Ja '65. (MIRA 18:5

1. Leningradskiy tekhnologicheskiy institut imeni Lensoveta.

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

[illegible] Part 9. Addi[illegible] of [illegible] and [illegible] analogs. Zhur. [illegible] (MIRA 18:5)

[illegible] Lensovet.

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"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

L 01047-66 EWT(m)/EPF(c)/EWP(j) RM

ACCESSION NR: AP5013771

UR/0366/65/001/005/0818/0821
543.51 : 547.317

24
30
B

AUTHOR: Polyakova, A. A.; Khmel'nitskiy, R. A.; Petrov, A. A.

TITLE: Mass spectra and structure of organic compounds. XII. Mass spectra and structure of disubstituted diyne hydrocarbons

SOURCE: Zhurnal organicheskoy khimii, v. 1, no. 5, 1965, 818-821

TOPIC TAGS: hydrocarbon, mass spectrum, dissociation

ABSTRACT: The article is a continuation of a study on the relationship between the electron impact and mass spectra of highly unsaturated compounds. In this work the mass spectra of three 2,4-diyne hydrocarbons differing in the structure of the saturated radical at the triple bond system were investigated: hexadi-2,4-yne, 6-methylheptadi-2,4-yne and 6,6-dimethylheptadi-2,4-yne. It was found that significant differences exist in ionization of the compared homologs. In the mass spectrum of hexadi-2,4-yne and its asymmetrical isomer, the molecular ion peak is the greatest. The fraction of total current due to molecular ions is about 31%. In dissociative ionization of the second and third hydrocarbons, the dissociation of molecular ions begins to play a much greater role. It was shown that the main direction of dis-

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"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

L 01047-66

ACCESSION NR: AP5013771

sociative ionization is splitting of the methyl radical. Further dissociation proceeds by splitting CH_2 and C_2H_2. It was shown that dissociative ionization during electron impact of diacetylene molecules which contain branched radicals in the α-position to the multiple bond system is analogous to the dissociative ionization of 1,3-enynes and 1,3-dienes with the same radicals. At the same time processes take place which are characteristic of the dissociation of diacetylenes. "The 6-methylheptadi-2,4-yne and 6,6-dimethylheptadi-2,4-yne were synthesized by Yu. I. Porfir'yeva and Ye. S. Turbanova." Orig. art. has: 1 table, 1 figure.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy institut po pererabotke nefti i gaza (All-Union Scientific Research Institute for Oil and Gas Refining); Leningradskiy tekhnologicheskiy institut imeni Lensoveta (Leningrad Institute of Technology)

SUBMITTED: 23May64 ENCL: 00 SUB CODE: OC

NO REF SOV: 006 OTHER: 001

Card 2/2

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

RADCHENKO, S.I.; PETROV, A.A.

Alkylthioenynes and their analogs. Part 5: Addition of lithium-alkyls to alkylthiovinylacetylenes. Zhur. org. khim. 1 no.6:993-996 Je '65. (MIRA 18:7)

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

BOGOLYUBOV, G.M.; MINGALEVA, K.S.; PETROV, A.A.

Dipole moments of certain acetylenic derivatives of phosphorus.
Zhur. ob. khim. 35 no.9:1566-1570 S '65. (MIRA 18:10)

1. Leningradskiy tekhnologicheskiy institut imeni Lensoveta.

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

MARKOVA, V.V.; KORMER, V.A.; PETROV, A.A.

Addition of organoboron compounds to 1,3-enyne hydrocarbons. Zhur. ob. khim. 35 no.9:1669-1672 '65. (MIRA 18:10)

1. Vsesoyuznyy nauchno-issledovatel'skiy institut sinteticheskogo kauchuka imeni S.V. Lebedeva.

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

L 59504-65 EPF(c)/EWP(j)/EWT(m) Pc-4/Pr-4 RM

ACCESSION NR: AP5013772 UR/0366/65/001/005/0822/0827

543.51:547.364

AUTHOR: Polyakova, A. A.; Popova, T. I.; Petrov, A. A.

TITLE: Mass spectra and structure of organic compounds. 13. Mass spectra of vinyl acetylene alcohol

SOURCE: Zhurnal organicheskoy khimii, v. 1, no. 5, 1965, 822-827

TOPIC TAGS: mass spectrum, vinyl acetylene alcohol, vinylacetylenylcarbinol, methylvinylacetylenylcarbinol, ethylvinylacetylenylcarbinol, dimethylvinylacetylenylcarbinol, ionization, dissociation, mass charge ratio, peak, intensity, peak intensity, ionization chamber, electron current

ABSTRACT: The mass spectra of vinylacetylenylcarbinol (1-pentene-3-yn-5-ol), methylvinylacetylenylcarbinol (1-hexene-3-yn-5-ol), ethylvinylacetylenylcarbinol (1-heptene-3-yn-5-ol), and dimethylvinylacetylenylcarbinol (5-methyl-1-hexene-3-yn-5-ol) have been studied. The results show that: 1) vinyl acetylene alcohols have a much higher resistance to electronic charges than saturated alcohols of the fatty series; 2) cleavage of the hydrogen or of the alkyl radical is the first stage in the dissociative ionization; 3) the formation of hydrocarbon ions is typical of

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"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

L 59504-65

ACCESSION NR: AP5013772

2

primary alcohols, and 4) the formation of oxygen-containing ions is typical of secondary and tertiary alcohols. A dissociation scheme for the maximal or intensive ions is suggested. The data on the mass spectra are given in Table 1 of the Enclosure. Orig. art. has: 1 figure and 2 tables.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy institut po pererabotke nefti (All-Union Scientific Research Institute for the Refining of Petroleum); Leningradskiy tekhnologicheskiy institut imeni Lensoveta (Leningrad Technological Institute)

SUBMITTED: 27Apr64 ENCL: 02 SUB CODE: OC,GP

NO REF SOV: 007 OTHER: 002

Card 2/4

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

L 59504-65

ACCESSION NR: AP5013772

ENCLOSURE: 01

Table 1. Mass spectra of vinyl acetylene alcohols

Mass-charge ratio, m/e	Intensity of peaks (in %) with respect to the maximal intensity				Mass-charge ratio, m/e	Intensity of peaks (in %) with respect to the maximal intensity			
	1-pentene-3-yn-5-ol (I)	1-hexene-3-yn-5-ol (II)	1-heptene-3-yn-5-ol (III)	5-methyl-1-hexene-3-yn-5-ol (IV)		1-pentene-3-yn-5-ol (I)	1-hexene-3-yn-5-ol (II)	1-heptene-3-yn-5-ol (III)	5-methyl-1-hexene-3-yn-5-ol (IV)
26	12.8	4.8	1.0	2.6	64	10.4	0.7	1.6	1.0
27	66.9	24.2	6.9	12.0	65	22.5	3.8	7.6	9.2
29	31.6	30.6	6.2	3.5	66	4.1	2.2	1.9	2.9
30	11.9	5.5	—	—	67	1.8	9.3	7.6	14.2
31	8.2	0.7	—	—	68	—	2.0	2.4	1.2
37	9.1	2.8	—	—	69	0.8	2.3	0.8	1.3
38	16.9	4.7	2.7	2.0	70	0.9	—	—	—

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APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

L 59504-65

ACCESSION NR: AP5013772 ENCLOSURE: 02

Table 1 (Cont.)

39	100.0	18.6	14.1	19.4	74	1.3	3.7	1.1	2.0
41	2.7	9.6	6.0	12.0	75	1.1	1.5	—	1.4
42	3.2	3.5	1.1	2.0	76	2.4	1.1	—	—
43	4.1	37.2	3.8	92.0	77	2.0	15.0	7.7	7.0
45	7.5	2.5	0.4	0.6	78	1.0	6.0	1.7	2.0
49	8.2	4.6	1.6	1.0	79	3.7	12.0	10.6	5.4
50	28.5	27.0	11.7	10.0	80	3.6	1.2	1.6	—
51	28.4	33.3	19.6	18.3	81	65.0	100.0	100.0	1.3
52	12.8	25.0	13.1	7.4	82	44.0	5.6	4.0	1.0
53	65.5	67.0	43.9	7.8	83	3.8	0.4	3.1	1.0
54	46.7	3.5	2.7	1.0	91	—	—	12.7	5.5
55	31.0	7.5	7.6	4.9	92	—	—	5.6	1.5
56	3.0	—	—	—	93	—	—	1.0	3.0
57	1.1	3.3	3.5	—	94	—	1.5	—	—
58	1.0	—	2.6	1.0	95	—	18.1	22.0	100.0
59	0.2	—	0.2	1.0	96	—	6.1	—	6.0
60	0.8	—	1.2	—	108	—	—	—	7.3
61	7.5	1.5	0.7	—	109	—	—	10.2	7.3
62	13.7	3.5	2.9	2.1	100	—	—	2.4	5.1
63	22.5	6.6	7.4	4.9					

Card KC 4/4

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LAZEYEVA, [illegible]; [illegible], A.A.; [illegible], L.A.

Use [illegible] for [illegible] in metals. [illegible] 1965.

(MIRA 18:4)

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L 56065-65 EWT(m)/EPF(c)/EWP(j) Pc-4/Pr-4 RM

ACCESSION NR: AP5018557 UR/0020/64/158/004/0907/0910

AUTHOR: Razumova, N. A.; Petrov, A. A.

20
B

TITLE: Condensation of glycolphosphorous acids chlorides with alpha, beta-unsaturated ketones

SOURCE: AN SSSR. Doklady, v. 158, no. 4, 1964, 907-910

TOPIC TAGS: condensation reaction, chloride, ketone

Abstract: Chlorides of glycolphosphorous acids were found to be capable of 1,4-addition with alpha, beta-unsaturated ketones, forming cyclic systems with two heteroatoms: isoxaphospholines. The condensation of the chlorides of ethylene-, propylene-, and 1,3-butyleneglycolphosphorous acids with methyl vinyl ketone and mesityl oxide produced products containing pentavalent phosphorus, which did not add oxygen and sulfur. The structure of the condensation products, as derivatives of isoxaphospholine, was confirmed by a study of the infrared and proton magnetic resonance spectra, and by the conversion of one of them (obtained from mesityl oxide) to 1-chloroisoxaphospholine oxide through the action of PCl_5. Orig. art. has 3 graph and 2 tables.

ASSOCIATION: Leningradskiy tekhnologicheskiy institut im. Lensoveta (Leningrad Technological Institue)

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ACCESSION NR: AP5018557

SUBMITTED: 08Apr64 ENCL: 00 SUB CODE: OC, GC

NO REF SOV: 005 OTHER: 004 JPRS

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BORISOVYEVA, Ye. I.; SOK[illegible] B.; [illegible] V. A. A.

Regularities in the [illegible] course of the addition of methylphenol [illegible] nearest homologues to diacetylene hom logs. Zhur. obs. khim. 34 no. [illegible] 1966 [illegible]

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[illegible]

Regularities [illegible]
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34 no.6[illegible]-128. [illegible]
.. Leningradskiy [illegible]

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PETROV, A. A.; [illegible] S.; [illegible] S.; SAVICH, I. [illegible]; [illegible] Y. B.

Alkyl thioethynes and their [illegible]. Part 1: Synthesis and properties of vinylacetylenic [illegible] selenos, and telluro ethers. Zhur. ob. Khim. 34 no. [illegible] (MIRA [illegible])

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YAKOVLEVA, T.V.; [illegible]; PETROV, A.A.

Vibrational spectra and the structure of organic compounds. Part 9. Opt. i spektr. 17 no. 4:[illegible] Ap '64. [illegible]

APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0"

"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

[illegible], A. A.; [illegible], [illegible] A.

"Metalation and isomerization of vi[illegible] the action of alkali metal amides [illegible]. [illegible] Khim. 34 no. [illegible]:1868-1873 [illegible] '64.

[illegible] Leningradskiy tekhnologicheskiy institut [illegible].

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"APPROVED FOR RELEASE: 07/19/2001 CIA-RDP86-00513R001240410009-0

PETROV, A.A.; FEDOROVA, A.V.

Allene hydrocarbons. Usp.khim. 3[illegible] no.1:3-27 Ja '64.
(MIRA 17:4)

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[illegible], A.A.; [illegible]

[illegible] cases of [illegible]
[illegible] no. [illegible]

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CA

The preparation of aromatic thiocyanates with the aid of dichlorourea. M. V. Likhashertov and A. A. Petrov. *J. Gen. Chem.* (U. S. S. R.) 8, 183-97 (1938).—Dichlorourea (I) and NH_4SCN react slowly in dil. aq. soln. giving pseudothiocyanogen (II), urea, H_2SO_4, HCN, HSCN and HCl. The formation of these substances may be explained by postulating the intermediate formation and hydrolysis of $CO(NHSCN)_2$ (III), the following reactions occurring: (1) $CO(NHCl)_2 + 2NH_4SCN \rightarrow CO(NHSCN)_2 + 2NH_4Cl$, (2) $CO(NHSCN)_2 + 2HOH \rightarrow CO(NH_2)_2 + 2HOSCN$, (3) $2HOSCN \rightarrow HSCN + NCSO_3H$, (4) $NCSO_3H + HOSCN \rightarrow HSCN + NCSO_3H$, (5) $NCSO_3H + H_2O \rightarrow HCN + H_2SO_4$. More concd. solns. of I and NH_4SCN react more quickly with evolution of heat, giving considerable quantities of II. In AcMe II is formed together with theoretical yields of urea and NH_4Cl. The reaction in MeOH is similar, yielding II, but in addn. a white substance, sol. in EtOH, insol. in H_2O, contg. much C, and exhibiting reducing properties. This unidentified substance reacts energetically with concd. HNO_3 with formation of free C. An Et_2O soln. of $Hg(SCN)_2$ (made by shaking $HgCl_2$ in Et_2O with NH_4SCN and filtering out the NH_4Cl) with the exactly theoretical quantity of I in Et_2O gave a transparent soln. free from I. This reacted with $PhNMe_2$ with pptn. of urea, furnishing evidence of III in the Et_2O soln. Likewise the red color of $Fe(SCN)_3$ in Et_2O is changed to the yellow of $FeCl_3$ by the addn. of I. III could not be isolated from its soln. in Et_2O or EtOH. All aromatic compds. contg. substituents favoring the introduction of halogen can be thiocyanated with I and NH_4SCN. The reaction occurring is $2RH + 2NH_4SCN + (NHCl)_2CO \rightarrow 2RSCN + 2NH_4Cl + (NH_2)_2CO$, since the urea and NH_4Cl can be quantitatively recovered. Phenols were best thiocyanated in aq. AcOH, amines in EtOH or aq. AcMe, and diamines and amino acids in AcOH or aq. EtOH. AcMe was especially suitable because most aromatic compds. and NH_4SCN are sol. therein, I acts upon AcMe only very slowly, and, since NH_4Cl is insol., the course of the reaction can be followed. The pptn. of orange II along with NH_4Cl indicates the absence of thiocyanation. A few drops of H_2SO_4 improve the quality and yield of product. Cooling and const. shaking during the introduction of I are required. $PhNMe_2$ (5.63 g.) and 3.54 g. NH_4SCN in 40 cc. AcMe with 3 g. I in 20 cc. AcMe gave, after filtration of the pptd. NH_4Cl, evapn. of the AcMe, and twice washing with alc., *p-dimethyl aminophenyl thiocyanate*, m. 71.4°. Recrystn. from EtOH or Et_2O did not raise the m. p. $PhNH_2$ (2.56 g.) and 2.5 g. NH_4SCN in 20 cc. of 70% MeOH, 1.5 drops of concd. H_2SO_4, and 2 g. I in 15 cc. of MeOH gave the sulfate of *p-aminophenyl thiocyanate* (IV), which was filtered and converted on the filter with Na_2CO_3 soln. to the free base IV (yield 80%), m. 56.7° after recrystn. With 2 mols. of I and NH_4SCN in AcMe, $PhNH_2$ gave

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80% of *2,6-dithiocyanoaniline*, m. 108° from EtOH or PhH. α-$C_{10}H_7NH_2$ (V) (4.4 g.) and 2.4 g. NH_4SCN in 20 cc. MeOH with 2 g. I in 10 cc. MeOH gave a product which was treated with large quantities of H_2O, whereupon 70-80% of readily crystallizable yellow oil formed. Recrystn. from aq. alc. gave *6-thiocyano-α-naphthylamine*, m. 146°. V (2.2 g.) and 2.36 g. NH_4SCN in 20 cc. AcMe with 2 g. I in 15 cc. AcMe gave *2,6-dithiocyano-α-naphthylamine*, decomp. 204°. NH_4Cl was filtered from the reaction mixt. while still warm and the filtrate on cooling gave needles of the above product which were recrystd. from EtOH. Addn. of H_2O to the mother liquor gave more product. L. and P. prepd. for the 1st time *4-thiocyano-* (VI), and *4,6-dithiocyano-m-phenylenediamine* (VII) and *dithiocyanobenzidine* (VIII). *p*-$C_6H_4(NH_2)_2$, because of its ready oxidizability, did not give a thiocyano deriv., and the monothiocyanobenzidine could not be sepd. from the accompanying oxidation products. *m*-$C_6H_4(NH_2)_2$ (1.08 g.) and 1.2 g. NH_4SCN in 20 cc. of 80% AcOH with 1 g. I in 10 cc. AcOH gave, after diln. of the reaction mixt. with H_2O, VI, m. 160-1° from MeOH, sol. in EtOH, Et_2O, AcMe and AcOH, insol. in H_2O and PhH. *m*-$C_6H_4(NH_2)_2$ with 2 mols. I and NH_4SCN gave VII, recrystd. from EtOH, m. 170-2° (decompn.), rhombic, less sol. than VI. VI (1.28 g.) and 1.84 g. KBr in 25 cc. 80% AcOH with 1 g. I in 15 cc. AcOH gave on cooling silvery crystals with a metallic luster, *4-thiocyano-6-bromo-m-phenylenediamine* (IX). The NH_2 groups in IX did not diazotize easily. IX decomposed on warming and was less sol. than VI. Benzidine (1.4 g.) and 1.2 g. NH_4SCN in 30 cc. AcOH with 1 g. I in 15 cc. AcOH gave, by pptn. from the reaction mixt. with H_2O and recrystn. of the ppt. from EtOH, VIII, decomp. 250°, difficultly sol. in EtOH, insol. in H_2O, sol. in AcOH and AcMe. *4-Thiocyanoanthranilic acid* (X) and *4-thiocyano-1-aminobenzene-3-carboxylic acid* (XI) were prepd. for the 1st time. Anthranilic acid (2.12 g.) and 1.2 g. NH_4SCN in 30 cc. AcMe with 1 g. I in 10 cc. AcMe gave on diln. 80% of X, recrystd. from hot H_2O, needles, m. 168-70°. X is insol. in cold H_2O, sol. in hot H_2O, EtOH, Et_2O, AcMe and hot AcOH. *m*-$NH_2C_6H_4CO_2H$, by a similar procedure, gave XI, decomp. on warming, insol. in H_2O, sol. in EtOH, Et_2O and AcMe. PhOH and $NO_2C_6H_4OH$ could not be thiocyanated with I and NH_4SCN. Hydroquinone and pyrocatechol were first converted to the corresponding quinones, then to the monothiocyanophenols, and finally to esters of thiocarbamic acid, $C_6H_4(OH)_2SCONH_2$. Resorcinol did not give a thiocyano deriv. α- (XII) and β-Naphthol (XIII) gave thiocyano derivs. with the same exptl. procedure. In the case of XIII there was formed at the same time a dark-red substance yielding on standing colorless needles of unknown compn. but not contg. a thiocyano group. *1-Thiocyano-β-naphthol* is readily decompd. with H_2O, giving a yellow solid which cannot be diazotized. XII (2.22 g.) in 20 cc. AcOH, 1.2 g. NH_4SCN in 5 cc. H_2O, and 1 g. of dry I, mixed in the order mentioned, gave on dilg. the mixt. with 3 parts of H_2O, 75% of *4-thiocyano-α-naphthol*, m. 112-13° from EtOH and H_2O, sol. in EtOH, Et_2O, difficultly sol. in AcMe, and insol. in H_2O. XII with 2 mols. NH_4SCN and I gave *2,4-dithiocyano-α-naphthol*, long yellow needles from EtOH and hot H_2O, m. 118° (decompn.), sol. in the common org. solvents. The yields of aromatic thiocyanates obtained by I and P are usually about 4-5 times those reported previously.

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ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION

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1ST AND 2ND ORDERS — PROCESSES AND PROPERTIES INDEX — 3RD AND 4TH ORDERS

CH

10

Halotxylation of unsaturated compounds. I. Hydroiodation and iodoalkoxylation of unsaturated compounds. A. A. Petrov. *J Gen. Chem* (U. S. S. R.) 4, 1217-23 (1934); cf. following abstr. MeCHICH(OH)Me (I), b_{10} ~ 84° (decompn.), d_4^{15} 1.798, n_D^{20} 1.539, was obtained by adding, with shaking, 3 mols. of powd. I in 3 portions to a mixt. of 1 mol. of freshly pptd. HgO in little H_2O and 1 mol. of 2-butene in Et_2O at −10° to −6°, sepg. from HgI, and H_2O, drying with $CaCl_2$, expelling the Et_2O at 55°, washing I with Na_2SO_3 soln. and storing in a dark bottle over $CaCl_2$. The product is practically free from impurities and can be reduced at reduced pressure at 65. The ethers of I were obtained with a mean yield of 80%, by adding powd. I to a mixt. of I and HgO in the alc. at −10° to −6°, shaking for a total of 10 min., filtering off the HgI, dilg. the filtrate with H_2O, sepg. the ether, washing with H_2O, distg. with steam in the presence of little dil. NaOH, drying with $CaCl_2$ and distg. For the prepn. of ethers from alcs. insol. in H_2O, the reaction was carried out in Et_2O. MeCHICH(OMe)Me, b_{760} 158.4°, b_{10} 53.4°, d_4^{15} 1.708, n_D^{20} 1.5026, gives on heating with dil. NaOH, AcONa and AgOH MeCHICH(OAc)Me, b_{10} 84.5°, d_4^{15} 1.5085, n_D^{20} 1.4955, and distg. with alc. NaOH or alcohol ates MeCH C(OMe)Me, b. 72.4°, d_4^{15} 0.8854, n_D^{20} 1.407, M. R. 28.31 (found), 28.4 (calcd.). MeCHICH(OEt)Me, b_{760} 170.2°, b_{10} 59.60°, d_4^{15} 1.5321, n_D^{20} 1.4906, gives on boiling for 6 hrs. with C_5H_5N in PhH MeCH NC$_5$H$_5$CH(OEt)Me, and with alc. NaOH MeCH C(OEt)Me, b. 40.3°, d_4^{15} 0.8885, n_D^{20} 1.4085, M. R. 30.43 (found), 30.6 (calcd.). Its isomer CH_2 C(OEt)CH$_2$Me was prepd. by Lauer and Spielman (C. A. 25, 2686). MeCHICH(OCHMe$_2$)Me, b_{10} 66-70.5°, d_4^{15} 1.4927, n_D^{20} 1.498, and MeCH C(OCHMe$_2$)Me, b. 106.8°, d_4^{15} 0.9211. MeCHICH(OCH$_2$CHMe$_2$)Me, b_{10} 79-82°, d_4^{15} 1.3775, n_D^{20} 1.481, and MeCH C(OCH$_2$CHMe$_2$)Me, b. 131.2°, d_4^{15} 0.8831, n_D^{20} 1.4188, M. R. 40.16 (found), 40 (calcd.). The stability and the fruity odor of the ethers decreases with the increasing mol. wt. of the alcs. The compds. of the type of MeCH C(OR)Me are produced for the first time, and are the ethers of the enol form of MeCOEt. Chas. Blanc

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Formation of phenols in the catalytic decomposition of ethanol by the method of S. V. Lebedev. M. V. Likhoshersto, A. A. Petrov and S. V. Alekseev. J. Gen. Chem. (U. S. S. R.) 4, 1274-80(1934). In the commercial production of divinyl by the catalytic decomposition of EtOH by the method of Lebedev (C. A. 28, 3050[9]), the "hydrocarbon" condensate contains varied proportions of homologous phenols, of which o-MeC$_6$H$_4$OH and o-EtC$_6$H$_4$OH were identified. Chas. Blanc.

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The halogenization of unsaturated compounds. II. The reaction of nitrogen triiodide with acids and alcohols in the presence of olefins. A. A. Petrov. *J. Gen. Chem.* (U. S. S. R.) 4, 1456-63(1934); *cf. C. A.* 29, 3905.—NI_3 reacts with H_2O and org. acids to give RCOOI (I) and with alcs. to give ROI (II). Addn. of I to an olefin is called iodoacyloxylation and addn. of II, iodoalkoxylation. The reaction was studied with 2-butene (III). Portions of moist NI_3 were added with cooling to the org. acid (contg. not more than 20% H_2O) and III cooled to −10°. The reaction occurring can be represented by: $NI_3.NH_3$ + 5RCO$_2$H + 3MeCH:CHMe → 2RCO$_2$NH$_4$ + 3MeCHI-CH(O CO.R)Me. At the end of the reaction, the products sep. into a lower org. layer and an upper aq. acid layer. After addn. of H_2O, the iodohydrin ester is washed with $Na_2S_2O_3$ and Na_2CO_3 soln. For solid acids Et_2O is used as a solvent. The esters so prepd. from III distil at 10-20 mm. almost without decompn. Any accompanying iodohydrin decomposes and distils first. The iodohydrin esters are very reactive. Alkalies convert them to oxides, Ag salts give esters of 1,2-glycols, and moist Ag_2O gives oxide, glycol and glycol monoester. Fuming HCl (30 cc.) cooled to −15° with 20 cc. III and NI_3 (from 26 g. I_2) gave 22 g. of *2-chloro-3-iodobutane* (IV), $b_{11.4}$ 49-52°, d_4^{15} 1.7771, n_D^{20} 1.5383. IV with alc. alkali gave a lachrymatory compd., probably 2-chloro-2-butene. *2-Bromo-3-iodobutane* (V), b_{16} 67-8°, d_4^{15} 2.0658, n_D^{20} 1.5730. V with alc. alkali gave first 2-bromo-2-butene and then dimethylacetylene. From H_2F_2 a substance was prepd., b_{16} 50°, d about 1.74, n_D^{20} 1.49, which was probably fluoroiodobutane. The following new *3-iodobutanes* were prepd.: *2-hydroxy*, b_{16} 84°, d^{16} 1.7980, n_D^{20} 1.5390; *2-formoxy*, b_{11} 72.5°, d^{16} 1.7597, n_D^{20} 1.5120; *2-acetoxy*, b_{16} 84°, d^{16} 1.5965, n_D^{20} 1.4955; *2-propioxy*, b_{16} 97°, d^{16} 1.4820, n_D^{20} 1.4858; *2-isobutyroxy*, b_{16} 101-9°, d^{16} 1.3996, n_D^{20} 1.4758; *2-isovaleroxy*, b_{16} 101-4°, d^{16} 1.3963, n_D^{20} 1.4820; *MeCHI-CH(OCH$_2$Ph)Me*, b_{16} 154-5°, d_4^{15} 1.4652, n_D^{20} 1.5610.

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Chloroalkoxylation of olefins with chlorine in the presence of alcoholic alkali. M. V. Likhosherstov and A. A. Petrov. *J. Gen. Chem. (U. S. S. R.)* 9, 1348-54(1939). C. A. 39, 3305, 3309, 8174[6]. Ethers of pseudo-butylene chlorohydrin were obtained in 60-70% yields with 25-35% of dichlorides as by-products by conducting 25% excess of Cl into a mixt. of 80% excess of powd. NaOH in an alc. at -5° with a gradual addn. of 2 liters of The reaction mixt. was diluted with H_2O until all NaCl was dissolved, and the sepd. layer of chlorohydrin ether was washed with H_2O to remove the unchanged alc. After drying with $CaCl_2$, the reaction mixt. was distd. to sep the ether from dichloride. MeCHCICH(OMe)Me, b. 118.8°, d^{20} 1.0102, n_D^{20} 1.426. MeCHCICH(OEt)Me, b. 122.6°, d^{20} 1.0882, n_D^{20} 1.424. MeCHCICH(OCH$_2$Ph)Me, b. 104.6°, d^{20} 0.9274, n_D^{20} 1.421. C. H.

ASTIA METALLURGICAL LITERATURE CLASSIFICATION

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1ST AND 2ND CODES

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH CODES

CA

Chloroalkoxylation of olefins. V. Pseudobutylene chlorohydrin ethers as solvents. M. V. Likhosherstov and S. V. Alekseev. *Acta Univ. Voronegiensis* 8, No. 2, 64-70(1935); cf. C. A. 32, 4524[6], 4525[1].—When these ethers are prepd. by the previously described methods, mixts. of the dichloride and β-Cl ethers are obtained which cannot be completely sepd. by distn. However, the mixts. can be used as solvents. The compds. studied have the formula MeCHClCH(OR)Me. When R is Me, the mixt. contains 27.6% dichloride, b. 116-21°, d^{20} 1.020, d^{10} 1.002, d^{20} 0.982, d^{40} 0.960, n_D^{20} 1.4262, n_D^{10} 1.4245; when R is Et, the mixt. contains 20% dichloride, b. 123-30°, d^{20} 1.0178, d^{10} 1.0082, d^{20} 0.9924, d^{40} 0.9684, n_D^{20} 1.4410, n_D^{10} 1.4292; the Pr mixt. contains 14.2% dichloride, b. 144-9°, d^{20} 0.9961, d^{10} 0.9848, d^{20} 0.9530, d^{40} 0.9342, n_D^{20} 1.4245, n_D^{10} 1.4218; the iso-Am mixt. contains 4.2% dichloride, b. 178-94°, d^{20} 0.9272, d^{10} 0.911, d^{20} 0.8850, d^{40} 0.8589, n_D^{20} 1.4320, n_D^{10} 1.4280. When R is Bu or iso-Pr the amts. of dichloride are not given, but the Bu mixt. b. 150-5°, d^{20} 0.938, d^{10} 0.928, d^{20} 0.911, d^{40} 0.891, n_D^{20} 1.4432, n_D^{10} 1.4320, and the iso-Pr mixt. b. 125-30°, [illegible], n_D^{10} 1.4298. The solubilities of the Me, Et and iso-Am mixts. at 0° are 0.270, 0.428 and 0.026 g. per 100 g. H_2O, resp. At 15° they are 0.190, 0.251 and 0.188, and at 30° they are 0.123 and 0.191 for the Me and Et compds. In stability, volatility and dissolving power the ethers compare favorably with the usual solvents. The corrosiveness of the lower homologs toward Cu, Pb and Fe is about that of $CaCl_2$, and increases as the mol. wt. of R rises. The Me and Et compds. mixed with alc. are good lacquer solvents. VI. **Synthesis and properties of β-hexylene chlorohydrin ethers.** A. A. Petrov and S. Ya. Fedoseev. *Ibid.* No. 4, 75-80. When β-hexylene, a by-product of the synthetic rubber industry, is treated in MeOH with dichloramine-T, it gives 20% *Me ether of β-hexylene chlorohydrin*, b. 159-61°, d_4^{20} 0.980, n_D^{20} 1.4475, M. R. 41.09. With chloramine-T and EtOH it gives the corresponding Et ether with 14% of the corresponding dichloride. The mixt. b. [illegible], d_4^{20} 0.9801, n_D^{20} 1.4481. When the Et ether is prepd. with dichloramine, the product contains 17% dichloride. The alkali method gives 48% and the NH_3 method 45% dichloride in the product. These mixts. are nearly as good solvents as the purer material which can be used in prepg. nitrocellulose lacquers. H. M. Leicester.

ASTM-ASA METALLURGICAL LITERATURE CLASSIFICATION

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

10

Ca

The reactions of conjugated systems with acyl derivatives. A. A. Petrov. *Acta Univ. Voronegiensis* 8, No. 2, 71 [illegible] *Zentr.* 1936, II, 2333-4; cf. C. A. 29, 3305. In the addn. of radicals to butadienes the point of entrance of the radical is deid. by the spatial relations between the size of the substituents and the configuration of the diene. In view of the smaller at. diam. of O as compared with that of the halogens, the addn. of HO compounds should take place predominately at the 1,2-position and not, as in the case of halogen compds., at the farther removed 1,4-position. This assumption was tested exptly. using the reaction between HOCl and butadiene, carried out as in C. A. 29, 3305. In addn. to some 1,4-dichloro-2-butene, very large amts. of 1-chloro-3-hydroxy-3-butene were formed, the constitution of which was established by detn. of the phys. properties and also by conversion into the butadiene 1,2-oxide. The variation between the phys. properties obtained for the prepn. and those given in the literature (cf. C. A. 27, 2673) was due to the presence of slight amts. of the oxide. The following ethers were prepd. from the oxide by boiling the latter 1 hr. with the alcs. in the presence of 1-2% Na alcoholate: *1-Methoxy-3-hydroxy-3-butene*, $C_5H_{10}O_2$, a hygroscopic liquid of faint odor and pungent, sweet taste, b. 161.4°, d_4^{20} 0.9043, d_4^{25} 0.9[illegible], d_4^{30} [illegible], n_D^{20} 1.4301, n_D^{25} 1.4263. R. Mr. [illegible]. The [illegible] obtained by treatment with AcCl, b. [illegible], d_4^{20} 1.1024, d_4^{25} 0.9993, d_4^{30} 0.9[illegible], n_D^{20} 1.4301, n_D^{25} 1.4208, R. Mr. 37.50. *1-Ethoxy-3-hydroxy-3-butene*, b. [illegible], d_4^{20} 0.9214, n_D^{20} 1.4430, R. Mr. 42.75. *1-Isobutoxy-3-hydroxy-3-butene*, b. 178 [illegible], d_4^{20} [illegible], n_D^{20} 1.4[illegible], R. Mr. 42.27. HOCl adds to butadiene contrary to Chloschenko and Petrov, C. A. 30, 2174; likewise in the 1,2-position instead of with the formation of the 1,4-ether of the chlorohydrin in addn. to the dichloride.

M. G. M.

ASTIA METALLURGICAL LITERATURE CLASSIFICATION

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The halogenation of aromatic compounds with *N*-chloro imidocarbonates. M. V. Likhosherstov, A. A. Ixtin and L. N. Voskresenskaya. *Acta Univ. Voronegensis* 8, No. 4, 57-62(1935).—*N*-Halogen derivs. of guanidine cannot be obtained in sufficient amts. for org. syntheses. When $(EtO)_2CNCl$ (I) reacts with $PhNH_2$, $PhNMe_2$ and o-C_6H_4OH, oxidation takes precedence over chlorination and tars are the chief products. However, a mixt. of I and o-$NO_2C_6H_4OH$ gives 60% of 4-chloro-2-nitrophenol. If KBr or KI is present in the mixt., the products are 4,6-dibromo- or 4-iodo-2-nitrophenol, resp. Chlorination of salicylic acid by this method gives 50.3% of the 5-chloro deriv., and with KBr present, the corresponding di-Br compd. is obtained. o-$NO_2C_6H_4NH_2$ under these conditions forms 62.8% of the 4-Cl, 72.7% of the 4-Br and 61.5% of the 4-I compd. The reactivity of I lies between that of dichlorourea and chloroacetamide. H. M. I.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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The. Iodoxylation of vinyl A A Petrov *Acta Univ Voronegiensis* 8, No 4, 63 7(1937) The addn of HOI to divinyl (I) in the presence of Ag_2O occurs in the 1,2-position, since supon. of the oxide prepd. from the reaction product gives $CH_2:CHCH(OH)CH_2OH$ When I, reacts in alc with I in the presence of HgO, only 1 of ROI adds even when a great excess of I, is used The amts of disulides formed as by-products increase with the mol wt of the alc used Addn always occurs in the 1,2-position and compds of the formula $CH_2:CHCH(OR)CH_2I$ are formed When R is *Me* the yield is 75% and the compd. b_{10} 65-5.5°, d_4^{16} 1.6110, n_D^{16} 1.5150, M R 30 68 When R is *Et*, the yield is 60% and the compd b_{10} 64-4.5°, d_4^{16} 1.5672, n_D^{16} 1.5108, M R 43 47 When R is *Pr* the yield is 35% and the compd b_{10} 63 75-4 5°, d_4^{16} 1.5504, n_D^{16} 1.5172, M R 46.50 When R is *iso-Bu* the compd. b_{10} 92-3°, d_4^{16} 1.3814, n_D^{16} 1.4950, M R 51 61 Alc KOH splits HI from these ethers to give the corresponding alkoxyprenes Br_2 adds to the Me deriv in glacial HOAc to give *1,2-dibromo-1-methoxy-4-iodobutane* b_{10} 150 1°, d_4^{16} 2.2121, n_D^{16} 1.5840, M R 45 25

H M Leicester

METALLURGICAL LITERATURE CLASSIFICATION

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PROCESSES AND PROPERTIES INDEX

CA

10

The synthesis and properties of alkoxypropenes. A. A. Petrov. *Acta Univ. Voronegiensis* 8, No. 1, 95-24 (1937). *C. A.* 32, 5370[9]. When butyral is treated in alc. with freshly ppted. HgO and I, at 10° and the product added to alc. KOH, alkoxypropenes of the formula $CH_3CHC(OR)CH_3$ are formed. The yields of Me and Et compds. are 52.5% based on the I, and of the other compds. somewhat less. The *Me compd.* b. 74.4°, d_4^{20} 0.8885, n_D^{20} 1.4480, n_∞ 25.10, M. R. 27.10, parachor 226.5. *Et compd.* b. 92.5-1.5°, d_4^{20} 0.8250, n_D^{20} 1.4480, n_∞ 25.20, M. R. 31.63, parachor 267.5. *Pr compd.* b. 118.6-19°, d_4^{20} 0.8190, n_D^{20} 1.4448, M. R. 36.30. *iso-Pr compd.* b. 105.7°, d_4^{20} 0.8117, n_D^{20} 1.4410, M. R. 36.40. *iso Bu compd.* b. 130.2°, d_4^{20} 0.8132, n_D^{20} 1.4450, n_∞ 24.51, M. R. 41.27, parachor 345.0. These compds. are easily hydrolyzed by H_2O to ROH and $CH_3CHCOMe$. The Me deriv. polymerizes 10 times less rapidly than chloroprene to a rubbery mass, but the polymers of the higher ethers appear to be dimers. All are polymerized by maleic anhydride to tars which become brittle when boiled with H_2O. H. M. Leicester

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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New methods of using olefins M. V. Likhosherstov, A. A. Petrov and S. V. Alekseev. *J. Chem. Ind.* (Moscow) 12, 60, 9(1935). - The com. possibilities of various methods of uniting esters of HOCl with double bonds are discussed, and the advantages of such compds. as MeCHClCH(OMe)Me as solvents are indicated.

H. M. Leicester

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The abnormal elimination of halogens from some tri- and tetra-substituted halogen compounds. A. A. Petrov and A. P. Sapozhnikova. *J. Gen. Chem.* (U. S. S. R.) 7, 476-84(1937). In compds. of the type RCHICX,R or RCBrXCX,R, when the halogens represented by X are more neg. than the I or Br (e. g., Cl), they cause an electron shift toward themselves, which permits the I or Br to sep. as a pos. ion. Therefore, when these compds. are treated with alc. KOH, a reductive splitting off of the I or Br occurs, with formation of hypohalite and an olefin, instead of the normal sepn. of hydrohalide. 2-Butene (I) adds Br_2 to form 2,3-dibromobutane which loses HBr with KOH to yield 2-bromo-2-butene (II). This brominates to 2,2,3-tribromobutane which with KOH goes entirely to 2,3-dibromo-2-butene (III). With Br_2 this forms 2,2,3,3-tetrabromobutane, which gives III again with alkali. Similarly, I and Cl_2 form 2,3-dichlorobutane. KOH converts this to 2-chloro-2-butene (IV), and IV with Br_2 gives *2-chloro-2,3-dibromobutane* (V), b. 182.5-6° (decompn.), b_{10} 99-6.5°, d_4^{20} 1.9146, d_4^{15} 1.8875, n_D^{20} 1.5369. KOH converts V into *2-chloro-3-bromo-2-butene* (VI), b. 128-9.5°, d_4^{20} 1.5202, d_4^{15} 1.4916, n_D^{20} 1.4930. VI and Br_2 give *2-chloro-2,3,3-tribromobutane* (VII), m. 22-3°. With KOH this yields VI again. A small amt. of the normal product, 2-chloro-3-bromo-1,3-butadiene, is probably present, since a resinous film sepa. on the walls after standing. IV and ICl give *2,2-dichloro-3-iodobutane* (VIII), $b_{10.4}$ 60.5°, d_4^{20} 1.9892, d_4^{15} 1.8580, n_D^{20} 1.5515. VIII and KOH form IV and, if not enough KOH is used, CH_3I. This shows the presence of KIO. II and ICl give a mixt. of the isomers MeCHICClBrMe and MeCHClCIBrMe, which cannot be sepd. KOH gives IV from the 1st and VI from the 2nd. The product of the union of II and IBr is a mixt. of MeCHICBrMe and MeCHBrCBrIMe (IX). With KOH these form II and III. From the relative amts. of the reaction products it appears that not less than 50% of the mixt. is IX. H. M. Leicester.

AVAILABLE METALLURGICAL LITERATURE CLASSIFICATION

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CA

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The emulsification processes taking place in the colloid mill. N. I. Kozin and A. A. Petrov. *Teoretsy Pitaniya* 7, No. 6, 60-64(1938); *Chem. Zentr.* 1940, 148; cf. *C. A.* 34, 3169.—By the use of a Russian colloid mill and by maintaining a definite ratio between the fat and water content, mayonnaise-like emulsions could be prepd. with a low (21%) or a high (67%) fat content. The stability of the emulsions was directly proportional to the r. p. m. and inversely proportional to the distance between the grinding stones. If the fat content is reduced or the water content is increased, the addn. of an emulsifying agent, increase in the content of dry substance, or an increase in the degree of dispersion is necessary. Under definite working conditions there is a max. for the amt. of emulsifying agent or of fat to produce a stable emulsion; if this max. is exceeded the emulsion becomes less stable. By the use of alk. casein as an emulsifying agent or by the addn. of dry albumin (11.1%) and with the further condition that the ratio between the solid and liquid phases be held at 1:2 or 1:3, emulsions can be produced the stability of which is independent of a reduction in the fat content (between 67 and 21%). M. O. Moore.

METALLURGICAL LITERATURE CLASSIFICATION

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Conjugated systems IV Interaction of divinyl with hypochlorous acid and its esters A A Petrov (Gen. Chem (U S S R) 8, 131-40 in French 141) (1938 cf C A 29, 3305 CH₂ CHCH(OH)CH₂Cl (I) was obtained when 1,3-butadiene (II 100% excess) was shaken with acidulated NH₂CONHCl at 12° for 30 min and the excess II was distd off at room temp (cf Lik homberatov and Akkaseev, C A 28, 8330 To obtain I free from the dichlorides insol in H₂O, the distn residue was extd with H₂O and the soln, after satn with NaCl, was extd with Et₂O The residue from the Et₂O when redistd gave 73% I (40% based on NH₂CONHCl), b 144 7°, d_4^{20} 1 1214, n_D^{20} 1 498, M R 29 58 29 4 calcd for C₄H₇OCl), acetate, b 183 6, d_4^{20} 1 1108, n_D^{20} 1 461 M R 35 96 On the addn of HClO to II the formation of 3 isomeric chlorohydrins is theoretically possible I, CH₂CICH CHCH₂OH (III) and CH₂ CHCHCICH₂OH (IV) I on digesting with 4 parts of 60% KOH gave CH₂ CH CH CH₂ O (V), an α oxide easily hydrolyzed to an unsatd glycol, and not the stable γ oxide O CH₂ CH CH CH₂ that would be derived from III I with 2 mols Br in CHCl₃ gave CH₂BrCHBrCH(OH)CH₂Cl and not CH₂BrCHBrCHCICH₂OH, derived from IV, since by oxidation with Na₂Cr₂O₇ + H₂SO₄ at 45° it gave CH₂Br CHBrCOCH₂Cl and no aldehydes or acids The dibromide, b₁₀ 129 30, d_4^{20} 2 0504, d_4^{20} 2 042, n_D^{20} 1 564, n_D^{20} 1 561, M R 42 44 (42 59 calcd The ketone, b₁₀ 132 5 d_4^{20} 2 0580, d_4^{20} 2 0521, n_D^{20} 1 556, n_D^{20} 1 554, M R 41 46 The results show conclusively that II adds HClO in the 1,2 position with the formation of only I V is highly reactive easily adding alcs with the formation of the hitherto unknown erythro monoethers CH₂CHCH OH CH₂Cl These were obtained by refluxing on a water bath for 12 hrs an equiv mixt of V and an alc with 1% of a Na alcoholate as a catalyst Under these conditions the danger of polymerization of V by the acid catalysts was eliminated Methoxy deriv yield b 145 4° d_4^{20} 0 947, n_D^{20} 1 436, n_D^{20} 1 4441 M R 27 99 28 03), acetate, b 159 d_4^{20} 1 [illegible] d_4^{20} [illegible] 1 420 n_D^{20} 1 [illegible] M R [illegible] 42 27 V with iso BuOH gave [illegible] Chloroalkoxy derivs of II of the general formula CH₂ CH OR CH₂Cl were obtained [illegible] with PhSiSiN₃ in small [illegible] Methoxy deriv [illegible] M R [illegible] 1 436 M R 27 99 28 03 Methoxy deriv [illegible] The ethers on heating with [illegible] KOH in an [illegible] gave [illegible] CH CHCOCH CH₂ [illegible] CH₂ CH OMe CH₂ [illegible] M R 31 41, and CH₂ CH OMe CH₂ [illegible] d_4^{20} 0 [illegible] n_D^{20} 1 44[illegible] M R 26 41 The [illegible] was proved when on dissolving in cold [illegible] H₂SO₄ the ether gave CH₂ CHCOMe, thus with PhNHNH₂ gave [illegible] methyl 3 pyrazoline [illegible] Thus, II adds alkyl hypochlorites in the 1,2 position The 1,2 ether with Br in CHCl₃ gave CH₂BrCHBrCH OR CH₂Cl, b₁₀ 142 [illegible]

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b_{10} 123-4°, d_4^{20} 1.7279, n_D^{20} 1.5182, M. R. 51.68. V.

Interaction of divinyl with hypobromous acid and acyl hypobromites. *Ibid.* 142-9(in French 150). CH_2:CHCH(OH)CH_2Br (VI) was prepd. in 52.5% yield on shaking II with acidulated $AcNHBr$ (from $AcNH_2$ and Br with HgO in H_2O) and proceeding as above. VI, b_{10} 56.5-7°, b. 161-2.5° (slight decompo.), d_4^{20} 1.6257, d_4^{20} 1.5205, n_D^{20} 1.5022, n_D^{20} 1.5, M. R. 29.21. VI with ArCl gave CH_2:CHCH(OAc)CH_2Br (VII) (65.5% yield), b_{10} 72°, d_4^{20} 1.3887, n_D^{20} 1.468, M. R. 38.65. VI with aq KOH gave 73% V, b. 66.5-7°, d_4^{20} 0.8764, n_D^{20} 1.4174, M. R. 20.13, EM_D 0.48. This with 2% H_2SO_4 gave erythrol. VI with Br in $CHCl_3$ at −10° gave 96.4% $CH_2BrCHBrCH(OH)CH_2Br$ (VIII), b_{10} 141-1.5°, d_4^{20} 2.3007, n_D^{20} 1.5556, M. R. 45.11. This (3.1 g.) on heating with 5 g. PBr_3 afforded 63.7% $CH_2BrCHBrCHBrCH_2Br$, m. 110.5° (alc.). VIII with ArCl gave 62.3% CH_2Br-$CHBrCH(OAc)CH_2Br$ (IX), b_{10} 140°, d_4^{20} 2.0649, n_D^{20} 1.5418, M. R. 54.48. VIII oxidized with CrO_3 gave 88% $CH_2BrCHBrCOCH_2Br$, b_{10} 121°, d_4^{20} 2.3214, n_D^{20} 1.577, M. R. 44.11. A mixt. of 0.5 mol. II and 1 mol. AcOH in 100 ml. Bu_2O was treated with 25 g. (1 mol.) $PhSO_2NBr_2$ at 20°. After washing with H_2O and 10% H_2SO_4, the Bu_2O was expelled and the residue redistd., giving 46% VII. This with aq. KOH gave V and with Br in $CHCl_3$ IX. Approx. 55 references. Chas. Blanc

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PETROV, A. A.

"Recherches dans le domaine des systemes conjugues. Communication V." Petrov, A. A. (p. 150)

SO: Journal of General Chemistry (Zhurnal Obschei Khimii) 1938, Volume 8, NO. 2

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Conjugated systems. The reaction of butyl with alkyl hypobromites and the synthesis of alkoxyprenes

A. A. Petrov. [illegible] (in French) 21[illegible] (1955). [illegible] preceding abstr. When solid $PhSO_2NBr_2$ is dropped into an alc. soln. of butyl alkyl hypobromite is formed and reacts to give CH_2 $CHCH(OR)CH_2Br$, where R depends on the alc. used. In this way are prepd. 54% of the compd. in which R [illegible] M_D [illegible] d_4^{20} 1.0[illegible] d_4^{20} 1.0[illegible] n_D^{20} 1.4[illegible] n_F^{20} 1.4[illegible] M R [illegible] of the *Et deriv.* b. 13[illegible] [illegible] d_4^{20} 1.2080, d_4^{20} [illegible], n_D^{20} 1.4018, n_F^{20} 1.4595, M R [illegible] 47% of the *Pr deriv.* b. 8[illegible] [illegible] d_4^{20} 1.2280, d_4^{20} [illegible] n_D^{20} 1.4[illegible], n_F^{20} 1.4092, M R 43.[illegible] 32% of the *Bu deriv.* b. 5[illegible] [illegible] d_4^{20} 1.1810, d_4^{20} [illegible] n_D^{20} 1.4[illegible], n_F^{20} 1.4[illegible], M R 48.[illegible] 30% of the *iso Bu deriv.* [illegible] M R [illegible] and 5[illegible] of the *sec Bu deriv.* b. 81-[illegible] d_4^{20} 1.1[illegible], d_4^{20} [illegible] n_D^{20} 1.4610, n_F^{20} 1.4[illegible], M R 52.45. Ph ethers cannot be prepd. in this way. These compds. lose ROHBr and regenerate butyl when they are treated with Zn dust or Mg. They are easily halogenated in cold $CHCl_3$ and thus give 78% of *3,4-dichloro-1-bromo-2-ethoxybutane*, bₚ 104-4.5°, d_4^{20} 1.4925, d_4^{20} 1.4965, n_D^{20} 1.4945, n_F^{20} 1.4962, M R 48.59; 92% of *1,3,4-trichloro-2-ethoxybutane*, bₚ 12[illegible], bₚ 142-3.5°, d_4^{20} 1.4490, d_4^{20} 1.4425, n_D^{20} 1.4[illegible], n_F^{20} 1.50.0, M R 54.45, and 95% of *1,3,4-trichloro-2-iso propoxybutane*, bₚ 13[illegible], d_4^{20} 1.8225, d_4^{20} 1.8449, n_D^{20} 1.5110, n_F^{20} 1.5202, M R 50.38. When the unsatd. ethers are treated with alc. KOH, they form the corresponding alkoxyprenes, CH_2:$CHCOR$ CH_2. In this way are prepd. 91.4% of the deriv. where R is Et [illegible] d_4^{20} 0.8240, d_4^{20} 0.81[illegible], n_D^{20} 1.4480, n_F^{20} 1.4490.

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Conjugated systems. VII. Synthesis and properties of 4,1-halo ethers of butadiene. A. A. Petrov. [illegible] Chem. [illegible] S. S. R. 8, 487-9; from French 407,[illegible]. [illegible] 1-4, 32, 5570. $CH_2BrCH:OR(CH:CR)CH_2Cl$ (I; R = alkyl) were prepd. in 80% yield from $CH_2BrCH(OR)CH:CH_2$ (II) (cf. loc. cit.) and Cl in $CHCl_3$ at 0°. The values given below are for b_{760}, d_4^{20}, d_4^{20}, n_D^{20}, n_D^{20} and M. R. (M. R. $_D$, resp.). I: R = *Me*, 97.7-5°, 1.5001, 1.5609, 1.5048, 1.5020, 44.01. *Et*, 104.4-5°, 1.4925, 1.4965, 1.4945, 1.4932, 48.79. *Bu*, 130°, 1.389, 1.3827, 1.4895, 1.4878, 57.8. *iso-Am*, 134.5°, 1.3424, 1.4558, 1.4890, 1.485, 62.32. II with Br as above gave 92-5% $CH_2BrCH(OR)CHBrCH_2Br$ (III): R = *Me*, 120.5°, 2.0832, 2.0738, 1.5522, 1.5498, 49.82. *Et*, 127°, 1.9166, 1.9425, 1.539, 1.537, 54.48. *Pr*, 137°, 1.8525, 1.8419, 1.5519, 1.520, 59.38. *Bu*, 149-0.5°, 1.7642, 1.7005, 1.5252, 1.5258, 63.6. *iso-Bu*, 141.5-5°, 1.751, 1.746, 1.5222, 1.52, 63.85. *iso-Am*, 154.5°, 1.6907, 1.6833, 1.52, 1.518, 68.48. I and III (0.1 mol.) with 1 g. hydroquinone in 25 ml. alc. react with 0.4 mol. of 25% KOH in alc., forming 62-83% $CH_2C(OR)CCl:CH_2$ (IV) and 55-65% $CH_2C(OR)CBr:CH_2$ (V), resp. (The values given below after the b. p. are for d_4^{20}, d_4^{20}, n_D^{20}, n_D^{20}, M. R. and BM_D, resp.) IV: R = *Me*, b_{760} 64-5°, 1.0715, 1.0669, 1.4734, 1.4706, 31.05, 0.19. *Et*, b_{760} 75-7°, 1.03, 1.0246, 1.4641, 1.4617, 35.51, 0.03. *Bu*, b_{17} 66.5-7°, 0.9934, 0.9883, 1.4663, 1.4645, 44.79, 0.07. *iso-Bu*, b_{17} 70.5-7°, 0.9754, 0.9728, 1.4652, 1.4625, 49.5, 0.16. V: R = *Me*, b_{20} 48.5-9.5°, 1.4170, 1.4116, 1.5068, 1.5011, 34.15, [illegible]. b_{20} 63.5-4°, 1.3100, 1.3055, 1.493, 1.4904, 39.24, [illegible]. *iso-Bu*, b_{20} 65.5-6°, 1.2984, 1.2946, 1.4852, 1.4805, 48.55, 0.9[illegible]. *iso-Am*, b_{20} 87.5°, 1.1618, 1.551, 1.4[illegible], 1.47[illegible], 51.51, 1.27. These compds. polymerize easily and when treated with cold 5% H_2SO_4 give ethyl chlorovinyl ketone and ethyl bromovinyl ketone, resp. Approx. [illegible] references.

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PETROV, A. A.

"Recherches dans le domaine des systemes conjugues. Communication VIII." Petrov, A. A. (p. 497)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii) 1938, Volume 8, No. 6

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Study of divinyl 1,3 butadiene [illegible] A. A. Petrov [illegible] 10, No 2, [illegible] summary in English [illegible] 32, [illegible] An extensive review on the methods of prepg 1,3 butadiene [illegible] and its deriva, with 300 references [illegible]

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The combined action of halo amides with acids on olefins. I. Synthesis of complex ester chlorohydrins of the isomeric butylenes. M. [illegible] Lukhoshevskiy and A. A. Petrov. *J. Gen. Chem.* (U.S.S.R.) 9, [illegible] (1939).

Halo amides react with acids in the presence of olefins predominantly in 2 directions, with the formation of β-Cl esters and free amides, and with the formation of chloroalkyl amides. It is highly probable that an unstable intermediate acyl hypochlorite is first formed: $PhSO_2NCl_2 + RCOOH \rightarrow PhSO_2NHCl + RCOOCl$. The synthesis of the ester chlorohydrins of pseudobutylene was effected by dissolving [illegible] mole acid in [illegible] butylene, and then [illegible] mole $PhSO_2NCl_2$ in [illegible] with cooling and shaking. The next day the excess acid was removed by washing the ether soln. with water and Na_2CO_3 soln. The residue on removal of the ether was fractionated at [illegible] mm. The [illegible] chlorohydrin $MeCHClCH_2OCH_2Me$, obtained in [illegible] yield, bp [illegible] 147.9°, bp [illegible] d_4^{20} [illegible] n_D^{20} 1.4417, MR [illegible] 42.16, [illegible] MR 0.28, [illegible] 1.4540, MR [illegible] bp 212.14°, bp [illegible] 41.27, *trichloroacetate* (80% yield), bp 124.5°, d_4 [illegible] n_D^{20} 1.4665, MR 51.40. With isobutylene, ether was omitted, and an excess of the acid served as a solvent. To a soln. of 18 cc. of liquid isobutylene and 0.5 mole acid was added 15.5 g. *N,N'*-dichlorourea (I). The mixt. was set aside at room temp., and shaken from time to time. The next day all of I had dissolved. The reaction mixt. was diluted with water, the oil sepd., and washed with water and Na_2CO_3 soln. On fractionating, considerable methylchloromethylethylene was isolated. The crude ester *chlorohydrin* of isobutylene, obtained in [illegible] yield, bp 140°, d_4^{20} 1.1136, n_D^{20} 1.4325, MR [illegible] 41.51, [illegible] (22%), bp 154°, d_4^{20} 1.1026, n_D^{20} 1.4340, [illegible] (5%), bp 102.5°, d_4^{20} 1.2270, n_D^{20} 1.4580, MR [illegible] 41.14, *trichloroacetate* (5%), bp 117.19°, d_4^{20} [illegible] n_D^{20} 1.4512, MR 51.14. **II. Combined action of benzenesulfondibromoamide with acids in the presence of pseudobutylene.** *Ind.* 31, [illegible] Better yields are obtained by using $PhSO_2NBr_2$ instead of the dichloride. The procedure is the same as for the dichloride. [illegible] above, the reaction is complete in 30-40 min. The [illegible] *bromohydrin* of pseudobutylene, $CH_3CHBrCH(OSOCHCH_3)$, obtained in 52% yield, bp 51.5° [illegible] d_4^{20} 1.4462, n_D^{20} 1.4610, MR 44.57, [illegible] 64.5, d_4^{20} 1.4413, n_D^{20} 1.4530, MR 50.27, [illegible] bp 106.5-10°, d_4^{20} 1.4015, n_D^{20} 1.4548, MR [illegible] 4.84, [illegible] 1.4925, MR 54.01, [illegible] n_D^{20} 1.4524, MR 48.01, [illegible] d_4^{20} 1.1001, n_D^{20} 1.4512, MR 51.12. H. Pavelka.

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Conjugated systems VIII Reaction of α chloro 1 3 butadiene with hypobromous acid and synthesis of chloro vinylethylene oxide

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Conjugated systems. IX. Reaction of 2-halobutadienes with alkyl hypoiodites and synthesis of halohydrins. A. A. Petrov. *J. Gen. Chem.* (U. S. S. R.) 10, 910-25(1940); *cf. C. A.* 34, 5050[9]. As had been shown previously, $CH_2CClCH:CH_2$ (I) reacts with HOBr with the formation of 4-bromo-2-chloro-1-buten-3-ol. In order to investigate whether the 3,4-addn. is a general reaction of 2-halobutadienes with hypohalogen derivs., the behavior of I and $CH_2CBrCH:CH_2$ (II) toward alkyl hypoiodites has been studied. The reaction products obtained correspond to the general formula $CH_2C(Hal)CH(OR)CH_2I$ since they yield compds. $CH_2C(Hal)C(OR):CH_2$ on treatment with alc. KOH soln., which are identical with previously described compds. This proves that the addn. has occurred exclusively in the 3,4-position. To 35.4 g. I in 75 ml. MeOH are added 44 g. HgO and 51 g. I, the latter in 5-g. portions, while cooling. HgI_2 and excess HgO are then filtered off, water is added to the filtrate and the ppted. oil is distd. in vacuo, whereby 62% (based on the I used) $CH_2CClCH(OMe)CH_2I$ (III), b_{10} 76.5-7°, d_4^{20} 1.7299, d_4^{20} 1.7135, n_D^{20} 1.5338, n_F^{20} 1.5312, is obtained. $CH_2CClCH(OEt)CH_2I$ (IV), b_{10} 82-3°, d_4^{20} 1.6231, d_4^{20} 1.6163, n_D^{20} 1.5220, n_F^{20} 1.5198, was obtained in an analogous manner in 46.2% yield. II (57 g.) and 51 g. I when reacted as described for III gave $CH_2CBrCH(OMe)CH_2I$ (V), b_{10} 91.5-2°, d_4^{20} 1.9402, d_4^{20} 1.9755, n_D^{20} 1.5586, n_F^{20} 1.5531, in 70% yield. $CH_2CBrCH(OEt)CH_2I$ (VI), yield about 47%, b_{10} 97.8°, d_4^{20} 1.9095, d_4^{20} 1.9512, n_D^{20} 1.5460, n_F^{20} 1.5435. III, IV, V and VI, on treatment with alc. KOH soln. gave $CH_2CClC(OMe):CH_2$, $CH_2CClC(OEt):CH_2$, $CH_2CBrC(OMe):CH_2$, and $CH_2CBrC(OEt):CH_2$, resp., identical with the compds. described previously (*C. A.* 34, 7091[1]).

X. Reaction of bromoprene with hypobromous acid. *Ibid.* 1013-20. — As was to be expected, $CH_2CBrCH:CH_2$ (VII) reacts with HOBr with the formation of $CH_2CBrCH(OH)CH_2Br$ (VIII), i. e., the addn. occurs again in the 3,4-position. VII (85 g.) on shaking at 0° with $CH_3BrCONH_2$ (contg. 0.4 g. mol. HOBr (obtained from 40 g. $AcNH_2$, 100 g. Br, and 104 g. HgO in 600 ml. H_2O) yields chiefly VIII, b_{10} 91.5-2°, d_4^{20} 2.0140, d_4^{20} 2.0100, n_D^{20} 1.5557, n_F^{20} 1.5535, and also smaller quantities of $CH_2BrCOCH(OH)CH_2Br$, bromides of VII and polymerization products of VII. VIII yields an *acetate*, b_{10} 99.5-100.5°, d_4^{20} 1.7872, n_F^{20} 1.5150, and on bromination with Br in $CHCl_3$ $CH_2BrCBr(OH)CH_2Br$ (IX), m. 91.5-3°. IX, on oxidation with $Na_2Cr_2O_7$ in dil. H_2SO_4 gives $CH_2BrCBr:COCH_2Br$, b_{10} 151-3°, d_4^{20} 2.5608, n_F^{20} 1.6120), which on reduction with Zn and 70% AcOH yields 2-butanone. When VIII (23 g.) is added dropwise to a 60% KOH soln. (contg. 25 g. KOH) heated at 150° there is obtained $CH_2CBrCH:CH_2O$ (X), (yield 66%), b_{10} 130.5-1°, d_4^{20} 1.5634, d_4^{20} 1.5756, n_D^{20} 1.4962, n_F^{20} 1.4936, which polymerizes on prolonged storage. X (7.4 g.) when shaken with 25 ml. 1% H_2SO_4

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The reactions of organic α-oxides with alcohols and carbonyl compounds in the presence of boron trifluoride. A. A. Petrov. *J. Gen. Chem.* (U. S. S. R.) 10, 981–96 (1940).—When cyclic oxides are heated in the presence of BF_3, polymerization occurs, and even when other reactions take place, polymerization is always a side reaction. The oxides in the presence of 0.015% BF_3 react with a 5-fold excess of EtOH or MeOH to give monoethers of diols. If less alc. is used, carbitol is also formed. With ketones, 1% BF_3 catalyzes the reaction with oxides below 40° to give cyclic acetals. Aldehydes react in a similar way, but inert solvents must be used to prevent too much polymerization. Acids react normally but acid chlorides in the presence of BF_3 cause only polymerization of the oxides. CH_2CH_2O (I) and MeOH give 80% $CH_2(OH)CH_2OMe$, b. 123.5–5°, d_4^{15} 0.9719, n_D^{20} 1.4042. With EtOH, I gives 80% $CH_2(OH)CH_2OEt$, b. 134–5°, d_4^{15} 0.9330, n_D^{20} 1.4090. Epichlorohydrin (II) and MeOH form 74.5% $ClCH_2CH(OH)CH_2OMe$, b. 170.5–1.5°, d_4^{15} 1.1093, d_4^{20} 1.1048, n_D^{20} 1.4498, n_D^{20} 1.4474, M. R. calcd. 28.83, found 28.59. Distn. of this over solid KOH gives glycide Me ether which reacts with MeOH and BF_3 to form $MeOCH_2CH(OH)CH_2OMe$, b. 168.5–9°, d_4^{15} 1.0139, d_4^{20} 1.0107, n_D^{20} 1.4228, n_D^{20} 1.4208, M. R. calcd. 30.10, found 30.18. Pseudobutylene oxide (III) and MeOH form 60% $MeCH(OH)CH(OMe)Me$, b. 133–5°, d_4^{15} 0.9124, n_D^{20} 1.4119. Isobutylene oxide (IV), b. 51.5°, d_4^{15} 0.8153, d_4^{20} 0.8117, n_D^{20} 1.3770, n_D^{20} 1.3745, M. R. calcd. 20.12, found 20.33, and MeOH give 75% $Me_2C(OMe)CH_2OH$, bₚₛ 141–1.5°, d_4^{15} 0.9365, d_4^{20} 0.9333, n_D^{20} 1.4208, n_D^{20} 1.4190, M. R. calcd. 28.46, found 28.17. The BF_3 must be removed by KOH before distn. of this compd. if decompn. is to be avoided. Some 4,4-dimethyl-2-isopropylidioxolane, b. 134–7°, is also formed in this reaction since iso-PrCHO, a by-product, reacts with IV in a side reaction. Cyclohexene oxide (V) and MeOH give 75% of the mono-Me ether of cyclohexanediol, b. 185–[illegible]°, d_4^{20} 1.0[illegible], d_4^{20} 1.0085, n_D^{20} 1.4620, n_D^{20} 1.4606. I and Me_2CO give 72% polymer of I and 12% 2,2-dimethyl-1,3-dioxolane, b. 92–2.5°, d_4^{15} 0.9460, d_4^{20} 0.9417, n_D^{20} 1.4020, n_D^{20} 1.4000, M. R. calcd. 26.38, found 26.27. With MeEtCO, I gives 19% *2-methyl-2-ethyldioxolane*, b. 118–18.5°, d_4^{15} 0.9408, d_4^{20} 0.9353, n_D^{20} 1.4130, n_D^{20} 1.4110, M. R. calcd. 30.99, found 30.77. The same compd. is obtained in 16% yield from glycol and MeEtCO with P_2O_5. I and MePrCO give 21.5% *2-methyl-2-propyldioxolane*, b. 140–40.5°, d_4^{15} 0.9293, d_4^{20} 0.9240, n_D^{20} 1.4198, n_D^{20} 1.4174, M. R. calcd. 35.61, found 35.42. II and Me_2CO give 59.3% 2,2-dimethyl-4-chloromethyldioxolane, bₚₛ 156.5–7.5°, bₚₛ 64.5–5°, d_4^{15} 1.1058, d_4^{20} 1.1008, n_D^{20} 1.4376, n_D^{20} 1.4352, M. R. calcd. 35.86, found 35.71. With MeEtCO, II forms *2-methyl-2-ethyl-4-chloromethyldioxolane*, bₚₛ 174–7°, bₚₛ 79–9.5°, d_4^{15} 1.0932, d_4^{20} 1.0887, n_D^{20} 1.4432, n_D^{20} 1.4412, M. R. calcd. 40.48, found 39.98. With MePrCO, II gives 84% 2-methyl-2-propyl-4-chloromethyldioxolane, bₚₛ 192–6°, bₚₛ 93.5–4.5°, d_4^{15} 1.0663, d_4^{20} 1.0625, n_D^{20} 1.4450, n_D^{20} 1.4427, M. R. calcd. 45.10, found 44.86. With PrCHO is obtained *2-propyl-4-chloromethyldioxolane*, bₚₛ 182–7°, bₚₛ 87.5–9.0°, d_4^{15} 1.0948, d_4^{20} 1.0924, n_D^{20} 1.4450, n_D^{20} 1.4430, M. R. calcd. 40.48, found 39.99. With the corresponding 3 ketones, the Me ether of glyceride gives up to 30% each of *2,2-dimethyl-3-methoxymethylidioxolane*, b. 154–5.5°, d_4^{15} 0.9809, d_4^{20} 0.9757, n_D^{20} 1.4150, n_D^{20} 1.4126, M. R. calcd. 37.25, found 37.20; *2-methyl-2-ethyl-3-methoxymethyldioxolane*, b. 171.5–3°, d_4^{15} 0.9803, d_4^{20} 0.9799, n_D^{20} 1.4223, n_D^{20} 1.4204, M. R. calcd. 41.87, found 41.53; and *2-methyl-2-propyl-3-methoxymethyldioxolane*, b. 188–91°, d_4^{15} 0.9686, d_4^{20} 0.9645, n_D^{20} 1.4260, n_D^{20} 1.4242, M. R. calcd. 46.49, found 46.07. III and PrCHO give 42.1% *2-propyldioxolane*, b. 156–7°, d_4^{15} 0.9042, d_4^{20} 0.9000, n_D^{20} 1.4165, n_D^{20} 1.4141, M. R. calcd. 40.28, found 40.04, and much polymer. With Me_2CO,

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[illegible] Reaction of [illegible] with [illegible] and [illegible] (V. Gen. Chem., [illegible], 1940, [illegible], 1013—1044). [illegible] (from NH(AcBr) [illegible] /10 mm. (acetate, [illegible] yield [illegible] b.p. [illegible]), which with [illegible] $CHCl_3$ gives [illegible] bromohydrin [illegible], b.p. [illegible]. This is oxidised (Na_2CrO_4 in H_2SO_4) to [illegible], b.p. 181—18[illegible]/10 mm. (II) [illegible] KOH yields bromopyruvic acid (III), b.p. [illegible] [illegible] H_2SO_4 12 hr. at 40°) [illegible] 121°/10 mm. (di-acetate, b.p. 110—111°/10 mm.). [illegible] Br in $CHCl_3$ (II) gives into β-bromo-[illegible] m.p. 121·5—123°, without with HBr [illegible] at —5° (II) affords [illegible] b.p. 90·5—101°/10 mm. (acetate, b.p. 100—100°/10 mm.). R. T

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PROCESSES AND PROPERTIES INDEX

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Conjugated systems XI Reaction of chloroprene with hydrogen bromide, the synthesis of ethers of 3-chloro-1-buten-1-ol A. A. Petrov. J Gen Chem (U S S R) 10, 1418-24(1940); cf. C. A. 35, 2112[illegible] and following abstr CH$_2$ CClCH CH$_2$ (85 g.) in 75 cc glacial AcOH at −5° was treated with 72 g gaseous HBr The reaction mixt was diluted with water and the oil which sepd was removed, washed with water and soda soln and dried over CaCl$_2$ The yield of *MeCCl CH CH$_2$Br* (I), b 100-2, d$_4^{20}$ 1.5335, d$_4^{0}$ 1.5204, nD 1.5185, nF 1.5100, MRD 33.51, was 72.5% (by wt of Br used) Oxidation of I with a slight excess of KMnO$_4$ yielded AcOH and BrCH$_2$CO$_2$H A mixt of 17 g I and 16 g Br in 50 cc CHCl$_3$ allowed to stay in ice water in diffused light for 24 hrs yielded *MeCCl BrCH BrCH$_2$Br*, bn 104.5-106°, d$_4^{20}$ 2.1807 Saponif of I with 10 parts soda soln by refluxing for 2 hrs yielded 80% of *3-chloro-crotonyl alc.*, MeCCl CHCH$_2$OH, b 101.2°, bn 78.5-79°, d$_4^{20}$ 1.1185, d$_4^{0}$ 1.1130, nD 1.4608, nF 1.4678, MRD 26.56 The alc was easily acetylated with AcCl, forming *3-chloro-crotonyl acetate*, bn 80.5-1.5°, d$_4^{20}$ 1.1078, d$_4^{0}$ 1.1020, nD 1.4517, nF 1.4605, MRD 30.10, and was also easily brominated, forming *3-chloro-2,3-dibromo-1-butanol*, bn 111-12.5°, d$_4^{20}$ 2.0145, d$_4^{0}$ 2.0158, nD 1.5005, nF 1.5544, MRD 42.53 Treatment of I with KOH in the corresponding alc gave the following ethers (their constants are given in the order bn, d$_4^{20}$, d$_4^{0}$, nD, nF, MRD), after refluxing for a short time (10-20 min) Me, the bromination of which yielded *MeCCl BrCH BrCH$_2$OMe*, bn 93.5°, 1.8110, 1.8280, 1.8230, 47.42. *Et*, Pr, 66.5-70°, 0.9744, 0.9680, 1.4440, 1.4410, 40.50, *Bu*, 80-78-80.5°, 0.9685, 0.9520, 1.4405, 1.4442, 45.23, *iso-Bu*, 80-81.5°, 0.9483, 0.9438, 1.4428, 1.4405, 45.42, *iso-Am*, 88-90.5°, 0.9447, 0.9304, 1.4409, 1.4449, 49.93; *allyl*, 73-73.5°, 1.0187, 0.9658, 1.4500, 1.4570, 40.10; *Ph*, 137.5-138.5°, 1.1150, 1.1117, 1.5301, 1.5170, 51.24, and *benzyl*, 180-180.5°, 1.0910, 1.0781, 1.5205, 1.5243 and 55.81 I easily reacted with NH$_4$CNS in cold MeOH, yielding the corresponding *thiocyanate*, bn 107.9°, d$_4^{20}$ 1.1882, nD 1.5608 All the compds obtained became yellow on standing, except the allyl and benzyl ethers which did not change at all A. A. Poderoms

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Conjugated systems. XII. Interaction of α-bromobutadiene with alkyl hypobromites. Synthesis of α-bromo γ-alkoxy derivatives of vinyl and of γ-alkoxyvinyl acetylenes. A. A. Petrov. *J Gen Chem* (U. S. S. R.) 10, 1883-8(1940); cf. preceding abst. CHBr:CHCH:OR)CH$_2$I (I) (R = alkyl) were prepd from CHBr:CHCH:CH$_2$, HgO, I and the corresponding alc., dried with $CaCl_2$ and reduced. In this way were prepd 70% of the compd in which R is *Me*, b$_{10}$ 80-90°, d$_4^{20}$ 2.0384, d$_4^{20}$ 2.0185, n$_D^{20}$ 1.5002, n$_F^{20}$ 1.5037, MR$_D$ 40.84. 70% of the *Et* deriv., b$_{10}$ 90-95°, d$_4^{20}$ 1.8898, d$_4^{20}$ 1.8888, n$_D^{20}$ 1.5020, n$_F^{20}$ 1.5054, MR$_D$ 51.16; 43.8% of the *Pr* deriv., b$_{10}$ 103.5-4°, d$_4^{20}$ 1.8073, d$_4^{20}$ 1.7990, n$_D^{20}$ 1.5444, n$_F^{20}$ 1.5420, MR$_D$ 55.74. I (0.1 mol.) and 0.2 g. hydroquinone in alc. react with 0.15 mol. KOH in alc., forming CHBr:CHC(OR):CH$_2$ (II) (low yields). R = *Me*, b$_{10}$ 57-8°, d$_4^{20}$ 1.4010, d$_4^{20}$ 1.3953, n$_D^{20}$ 1.5170, n$_F^{20}$ 1.5145, MR$_D$ 35.18. *Et*, b$_{10}$ 69-71°, d$_4^{20}$ 1.3886, d$_4^{20}$ 1.3837, n$_D^{20}$ 1.5028, n$_F^{20}$ 1.5000, MR$_D$ 40.03. I (0.1 mol.) and 0.5 g. hydroquinone in alc. react with excess KOH (1.0 mol.) in 100 ml. alc., forming CH:CC(OR):CH$_2$ (III). R = *Me* (IV), b$_{10}$ 87-7.5°, d$_4^{20}$ 0.8770, d$_4^{20}$ 0.8718, n$_D^{20}$ 1.4402, n$_F^{20}$ 1.4440, MR$_D$ 24.00 (yield 42.0%). *Et*, b$_{10}$ 103.5-4°, d$_4^{20}$ 0.8666, d$_4^{20}$ 0.8621, n$_D^{20}$ 1.4472, n$_F^{20}$ 1.4446, MR$_D$ 29.93; *Pr*, b$_{10}$ 124-5°, d$_4^{20}$ 0.8508, d$_4^{20}$ 0.8383, n$_D^{20}$ 1.4484, n$_F^{20}$ 1.4460, MR$_D$ 34.30. CH:CCOCH$_3$, obtained in 2.5-g. yield by shaking 5 g. IV with 50 ml. H_2SO_4 at 35-40°, satg. with $(NH_4)_2SO_4$, and extn. with Et_2O, b$_{10}$ 83.5-4°, d$_4^{20}$ 0.8900, d$_4^{20}$ 0.8703, n$_D^{20}$ 1.4080, n$_F^{20}$ 1.4070, MR$_D$ 18.08. S. Kaganoff

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PROCESSES AND PROPERTIES [illegible]

Conjugated systems. XIII. The reaction of 1-bromobutadiene and hypobromous acid. The synthesis and properties of 1-bromobutadiene oxide. A. A. Petrov. *J. Gen. Chem.* (U. S. S. R.) 10, 1887-91(1940). *C. C.* 35, 4503*. Bromination of bivinyl gives 52% 1,4-dibromo-2-butene, m. [illegible]. With powd. KOH this gives [illegible]% CHBr:CH:CH:CH$_2$, b. [illegible], d_4^{20} 1.0827, n_D^{20} 1.5134, n_α^{20} 1.5100, n_γ^{20} 1.5168, *MR* calcd. [illegible], found [illegible]. When this is shaken with an acid soln. of bromoacetamide, it adds HOBr to form 1,4-dibromo-3-buten-2-ol (I). This 3,4-addn. confirms the work of Ingold and Smith (C. A. 26, 602) but contradicts that of Muskat and Grimsley (C. A. 27, 2834). I, b. [illegible], d_4^{20} 2.0222, d_4^{20} 2.0102, n_α^{20} 1.5602, n_γ^{20} 1.5651, *MR* calcd. 47.26, found 30.71, has a soly. at 15° of about 3 g. per [illegible] H_2O. With AcCl I forms 80% of the *acetate*, b. [illegible], d_4^{20} 1.7708, d_4^{20} 1.7782, n_α^{20} [illegible], n_γ^{20} [illegible], *MR* calcd. 40.02, found 40.26. With Br_2 I gives 75.2% *1,1,4,4-tetrabromo-2-butanol*, b. [illegible], d_4^{20} 2.0262. $NaClO_3$ in HOAc oxidizes this to give [illegible] of the [illegible] *bromo-butanone*, m. [illegible]. This is reduced by [illegible] and HOAc to MeBrCO. When I is dropped slowly into 60% KOH, it gives 30.2% *1-bromobutadiene oxide* (II), b. [illegible], b$_{760}$ 145.0°, d_4^{20} 1.0205, d_4^{20} 1.0114, n_α^{20} [illegible], n_γ^{20} 1.5100, *MR* calcd. 27.41, found 27.60. Treatment of II with HBr forms CHBr:CH:CHBrCH$_2$OH, b. [illegible], d_4^{20} 2.0245, n_D^{20} 1.5081. II dissolves in 5% H_2SO_4 to form 63% *4-bromo-3-butene-1,2-diol*, b$_{10}$ 120.5°, d_4^{20} 1.6082, d_4^{20} 1.6037, n_α^{20} 1.5300, n_γ^{20} 1.5280, *MR* calcd. [illegible], found 30.52. II is more reactive to H_2O, has a greater tendency to polymerize, and loses HBr to form the acetylene oxide more easily than bromoprene oxide.

H. M. Leicester

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Conjugated systems XIV The condensation of diene hydrocarbons with methyl vinyl ketone Synthesis and properties of 1,2,3,6-tetrahydroacetophenone and its derivatives A. A. Petrov. *J Gen Chem* (U S S R) 11, 330-13 (1941), cf *C A* 35, 4347[7]. When CH_2CH-$COMe$ (I) is heated with butyl in a sealed tube at 140° for 8-10 hrs, it gives 75-80% *1,2,3,6-tetrahydroacetophenone*, bₚ 184-5°, bₚ 70.5-80°, d_4^{20} 0.9829, d_4^{20} 0.9584, n_D^{20} [illegible], n_D^{20} 1.4608, *MR* calcd 38.49, found 38.09 [illegible] 1.4720, n_D^{20} 1.4608, *MR* calcd 38.49, found 38.09 [illegible] *semicarbazone*, m 165-6°, *p-nitrophenylhydrazone*, m 142-[illegible] With isoprene, I gives 75-80% 4-methyl-1,2,3,6-tetrahydroacetophenone, bₚ 204.5-6°, bₚ 94.2-4.5°, d_4^{20} 0.9446, d_4^{20} 0.9408, n_D^{20} 1.4725, n_D^{20} 1.4704, *MR* calcd 41.11, found 41.02, *semicarbazone*, m 151°, *p-nitrophenylhydrazone*, m 166-8°. This compd may contain traces of an isomer. I and CMe_2CH_2 give 75-80% *4,5-dimethyl-1,2,3,6-tetrahydroacetophenone*, bₚ 218-20°, bₚ 108.5-9°, d_4^{20} 0.9420, d_4^{20} 0.9292, n_D^{20} 1.4701, n_D^{20} 1.4774, *MR* calcd 45.72, found 45.28, *semicarbazone* m 181-2°, *p-nitrophenylhydrazone* m 159-72°. With 1,3-cyclohexadiene, I gives 50% *1,6-endoethylene-1,2,3,6-tetrahydroacetophenone*, bₚ 139-[illegible], d_4^{20} [illegible], d_4^{20} 1.0152, n_D^{20} 1.4958, n_D^{20} 1.4938, *MR* calcd [illegible], found 43.01, *semicarbazone*, decomps 185-[illegible], *p-nitrophenylhydrazone*, m 120-0.5°. The crystal forms of the 4-*p*-nitrophenylhydrazones differ greatly. H. M. [illegible]

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Conjugated systems. XV. The condensation of alkoxyprenes with acrolein, synthesis of 1-formyl-4-cyclohexanone and its derivatives. A. A. Petrov. *J. Gen. Chem.* (U. S. S. R.) 11, 601-4(1941); cf. *C. A.* 35, 5873[8].

When $CH_2C(OMe)CH:CH_2$ (I) is heated in a sealed tube at 120-40° for 6 hrs. with acrolein in the presence of a little hydroquinone, it gives 65% *1-formyl-4-methoxycyclohexene* (II), $b_{10.02}$ 92-2.5°, d_4^{20} 1.0563, d_4^{20} 1.0521, n_D^{20} 1.4829, n_D^{20} 1.4880, *MR* calcd. 38.13, found 37.87. Solv. and acrylic acid must be absent from the reaction mixt., or I will polymerize completely. An analogous reaction gives 50% *1-formyl-4-ethoxycyclohexene*, b_{10} 101.5-2°, d_4^{20} 1.0210, d_4^{20} 1.0175, n_D^{20} 1.4770, n_D^{20} 1.4750, *MR* calcd. 42.75, found 42.65. These compds. tend to polymerize and are especially easily hydrolyzed. When II is shaken with 1-2% H_2SO_4, it forms MeOH and *1-formyl-4-cyclohexanone* (III), b_{10} 113-3.5°, d_4^{20} 1.0953, d_4^{20} 1.0900, n_D^{20} 1.4780, n_D^{20} 1.4760, *MR* calcd. 32.35, found 32.58; *disemicarbazone*, m. about 199°; *bis-p-nitrophenylhydrazone*, orange powder). On standing, III polymerizes to crystals, m. 255°. When III is oxidized with $KMnO_4$, it gives *p*-cyclohexanonecarboxylic acid whose hydrate, m. 160-7°, has anisotropic crystals.

H. M. Leicester

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PROCESSES AND PROPERTIES INDEX

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Conjugated systems XVI Condensation of diene hydrocarbons with unsaturated aryl ketones N. A. Natsinskaya and A. A. Petrov. *J. Gen. Chem.* (U. S. S. R.) 11, 665-8(1941); cf. *C. A.* 35, 5873[9]. — The authors investigated the condensation of diene hydrocarbons (bivinyl and isoprene) with a no. of unsatd. aryl ketones. PhCH:CHCOMe (7 g.) and 10 cc. bivinyl heated at 170-80° (sealed tube) for 10 hrs. yielded 47% of *5-phenyl-4-acetylcyclohexene*, m. 62.2-62.7° (from EtOH); *oxime*, from the ketone and NH_2OH in EtOH pptd. by addn. of H_2O, m. 94.5-95° (from EtOH); *semicarbazone*, from the ketone and semicarbazide-HCl in EtOH-H_2O in the presence of KOAc, m. 175-6° (from EtOH). PhCH:CHCOPh (10 g.) and 8 cc. bivinyl heated (sealed tube) 10 hrs. at 100-80° gave 82% *5-phenyl-4-benzoylcyclohexene*, m. 100.4-1.5° (from EtOH); *dibromide*, m. 120.2-1.2° (from MeOH). PhCH:CHCOPh (2 g.) and 3 g. isoprene heated (sealed tube) for 10 hrs. at 150° yielded 50.6% *5-phenyl-4-acetylmethylcyclohexene*, m. 105.6° (from EtOH); *dibromide*, m. 111.5-12° (from EtOH); use of an excess of Br yields a substance, m. 153-4.5°, $C_{16}H_{16}OBr$. Dibenzalacetone (2.3 g.) and 2 cc. bivinyl heated (sealed tube) for 10 hrs. at 170-80° yielded *7,2'-diphenyl-1,1'-2,2',3,1',6,6'-octahydrobenzophenone*, m. 164.5-4.7° (from EtOH); *dibromide*, m. 215.6° (from $CHCl_3$). In contrast to phenylacetylcyclohexene, the phenylbenzoylcyclohexenes do not react with derivs. of N_2H_4.

G. M. Kosolapoff

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Action of acetyl chloride and bromide in halogenated α-alkyl-

A. A. [illegible] (*J. Gen. Chem. Russ.*, 1941, 11, 715—721).—α- and α'-methylpropylchlorohydrins and the corresponding bromohydrins undergo ring fission with H halides, giving rise to alcohols CHMeHal-CH(OH)-CH_2Hal. Crotyl bromide, b.p. 104·5—106·5°, is hydrolysed to a mixture of OH·CHMe·CH:CH_2, b.p. 95—97°, and CHMe:CH·CH_2·OH, b.p. 121—122°, separated by fractionation. Addition of Br in $CHCl_3$ affords, respectively, *γδ-dibromobutan-β-ol*, b.p. 94·5°/10 mm. (*acetate*, b.p. 105—106·5°/10 mm.), and *βγ-dibromobutan-α-ol*, b.p. 99·5°/10 mm. (*acetate*, b.p. 100·5°/10 mm.). With aq. KOH these yield *α-methyl-α'-bromomethylethylene oxide* (I) (70%), b.p. 144—145·5°, 64·5—65°/25 mm., and *α-bromomethylethylene oxide* (30%), evidently a mixture of two stereoisomerides, the main fraction (II), b.p. 142—145°, and (III), b.p. 152—154°; these are accompanied by (?) divinyl oxide, b.p. 62—65°. (II) with fuming HCl yields *α-chloro-γ-bromobutan-β-ol* (IV), b.p. 76—76·5°/10 mm. (*acetate*, b.p. 92·5—95°/10 mm.), oxidised to α-chloro-γ-bromobutanone, b.p. 67—68°/10 mm. (II) with HBr gives *αγ-dibromobutan-β-ol* (V), b.p. 90·5—91°/10 mm. (*acetate*, b.p. 105—106°/10 mm.); (III) gives an isomeride, b.p. 90·5—91°/10 mm.; both are oxidised to the same *αγ-dibromobutanone*, b.p. 76·5—77°/10 mm.; with KOH both give the same oxide (I). (I) with HCl affords *γ-chloro-α-bromobutan-β-ol* (VI), b.p. 76·5—77°/10 mm. (*acetate*, b.p. 94·5—96·5°/10 mm.), oxidised to *γ-chloro-α-bromobutanone*, b.p. 64·5—65°/10 mm. (I) with HBr gives (V). (IV) with KOH gives *α-methyl-α'-chloromethylethylene oxide* (VII), b.p. 124—125·5°; (VI) similarly gives *α-chloroethylethylene oxide* (VIII), b.p. 118—124°, and a smaller amount of an *isomeride*, b.p. 128—130°. (VIII) with HCl gives *αγ-dichlorobutan-β-ol* (IX), b.p. 63—64°/10 mm. (*acetate*, b.p. 69·5—70·5°/10 mm.); the higher-boiling isomeride of (VIII) gives a product, b.p. 63—64°/10 mm.; both are oxidised to αγ-dichlorobutan-β-one, b.p. 55—55·5°/10 mm. (VIII) with HBr gives (VI). (VII) with HCl gives (IX) and with HBr, (IV). All the halogenated ketones are reduced with Zn and AcOH to COMeEt.

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Conjugated systems. Avram. Reaction of chloroprene with chlorine. A. A. Petrov. *J. Gen. Chem.* (U.S.S.R.) 13, 103-7(English summary, 107)(1943); *cf. C. A.* 37, 1660*.—P. investigated chlorination of chloroprene in continuation of his long study of unsatd. compds. When chloroprene (155 g.) and 1 g. hydroquinone in 1 l. $CHCl_3$ were cooled to −10° and, with vigorous stirring, treated with 83 g. Cl in 500 cc. $CHCl_3$, the temp. being kept between −5° and −10° there is evidence of HCl evolution especially at the end of the reaction. The product after washing was distd. *in vacuo*. Chloroprene dichlorides (b_{10} 34-70°) amounted to 70% of the total products. The main fraction (b_{10} 66-70°) on redistn. gave *1,3,4-trichloro-1-butene*, b_{10} 67-9°, d_4^{15} 1.2675, n_D^{15} 1.5121; treated with $KMnO_4$, it gave 72% $ClCH_2CO_2H$, thus confirming the structure of this isomer. A fraction, b_{10} 40-2°, d_4^{15} 1.3190, n_D^{15} 1.4902, on oxidation as usual, gave $ClCH_2CHClCO_2H$, on the basis of which it appears to be *1,2,3-trichloro-1-butene*. The lowest-boiling fraction, on further washing and redistn., gave *1,2-dichloro-1,3-butadiene*, b_{10} 48-8°, d_4^{15} 1.1905, n_D^{15} 1.5065. The distn. residue of the crude reaction mixt. amounted to 30% of the total and contained less Cl than necessary for trichlorobutenes. 1,2,4-Trichloro-2-butene (64 g.) contg. 0.2 g. hydroquinone, slowly added to 24 g. KOH in 150 cc. boiling EtOH, and the mixt. steam-distd. yielded 42 g. crude dichlorobutadiene, which redistd., gave 36 g. pure 1,2-dichloro-1,3-butadiene, b_{10} 46.75-7.5°, d_4^{15} 1.199, n_D^{15} 1.5078; there was also formed a small amt. of material b_{10} 75-85°, apparently an EtO deriv., which was not studied further. XIX

Bromination of chloroprene. *Ibid.* 109-12(English summary, 112).—Chloroprene (26 g.) in 100 cc. $CHCl_3$ was cooled to 0-5° and treated with stirring with 40 g. Br in 50 cc. $CHCl_3$; after removal of the $CHCl_3$, the reaction mixt. was distd. *in vacuo*. 5.5 g. b_{10} below 98° and 48.5 g. b_{10} 98-101°; the latter was the *chloroprene dibromide*, which, oxidized with alk. $KMnO_4$, gave $BrCH_2CO_2H$, showing its 1,4-structure. If the bromination is carried out in the presence of antioxidants, the distn. range of products is much wider, apparently due to the formation of 2 geometrical isomers; all fractions on oxidation gave $BrCH_2CO_2H$. EtOH (100 cc.), 25 g. Zn and 0.5 g. hydroquinone heated to boiling were treated dropwise with 50 g. of the above chloroprene dibromide with simultaneous distn. of lower-boiling reaction products; the latter represented, on redistn., 75% chloroprene yield. Alc. KOH, treated with chloroprene dibromide (25 g.) added dropwise, followed by refluxing for 0.25 hr. and steam-distn., yielded chlorobromobutadiene, b_{10} 65-7.5° (0.2 g.), and *1,4-diethoxychloroprene*, b_{10} 75-85°, contaminated with ethoxybromochloroprene (1.4 g.). ($CHCl_2Br$), treated analogously with alc. KOH gave *1,4-diethoxy-2-butene*, b_{10} 61.75-2.5°, d_4^{15} 0.9099, n_D^{15} 1.4220, and a small amt. of 1-bromobutadiene; bromination of the di-EtO compd. in $CHCl_3$ gave a cryst. *bromide*, m. 28-30°, b_{10} 119.5-20.5°.

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PETROV, A. A.

"Conjugated systems. XIX. Bromination of chloroprene." Petrov, A. A. (p. 112)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii) 1943, Volume 13, no. 1-2.

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Conjugated systems. XX. Reaction of chloroprene with ICl. A. A. Petrov. *J. Gen. Chem.* (U. S. S. R.) 13, 165-8(1943)(English summary). cf. *C. A.* 38, 3298.
The author studied the addn. of ICl to chloroprene. Chloroprene, stabilized with pyrogallol, in $CHCl_3$ was treated at −5° to 0° with ICl in $CHCl_3$. The reaction mixt. was washed with hyposulfite and distd. in vacuo; the over-all material bo. 75-105°. In the absence of stabilizer the resulting mixture was similar, but considerable amts. of chloroprene polymer resulted. Oxidation of the crude product with $KMnO_4$ yielded only $ClCH_2CO_2H$. The crude product (52.2 g.) was treated with a boiling soln. of 25 g. KOH in 100 cc. EtOH, in the presence of hydroquinone, to yield 2 g. chloroprene and 10.5 g. *1,2-dichloro-1,3-butadiene*, bo. 40-4°. On the basis of previous experience in such dehalogenations it is believed that addn. of ICl to chloroprene results in formation of 70% 1,4 addn. product to give *1,4-dichloro-1-iodo-2-butene*. **XXI. Reaction of 1,2-dichloro-1,3-butadiene with HBrO and alkyl hypoiodites. Synthesis and properties of dichlorovinylethylene oxide and 1,2-dichloro-3-alkoxy-1,3-butadienes.** *Ibid.* 230-6(English summary). 1,2-Dichloro-1,3-butadiene was stirred with the calcd. amt. of acidified (1% H_2SO_4) 10% aq. soln. of bromosuccinamide to yield a bromohydrin *CHCl:CCl:CH(OH)CH₂Br* (65%), bo. 100-12°, *d₄* *deriv.* (by AcCl), bo. 109-12°. Dehydr. of the bromohydrin from 80% KOH gave the oxide, *CHCl:CCl:CH:CH₂O* (70%), bo. 94-5.5°, d_4^{20} 1.3724, n_D^{20} 1.5010, which on stirring at 60° with 5% H_2SO_4 gave the glycol, *CHCl:CCl:CH(OH)CH₂OH* (90%), bo. 135-6°, d_4^{20} 1.4580, n_D^{20} 1.5108, which treated with AcCl gave the *di-Ac deriv.* (75%), bo. 128.5-9°, d_4^{20} 1.2977, n_D^{20} 1.4802. The bromohydrin after bromination in $CHCl_3$ and oxidation by $Na_2Cr_2O_7$ yielded a ketone which on reduction by Zn in AcOH yielded 70% $MeBrCO_2$, thus confirming the bromohydrin structure given above. The dichlorobutadiene treated with aniline and HgO in MeOH gave *CHCl:CCl:CH(OMe):CH₂* (60%), bo. 90-1.5°, d_4^{20} 1.0147, n_D^{20} 1.5032. Similarly, the *Et deriv.* was prepd., which is 94-5.5° (decompn.). On boiling with 20% KOH these yielded the corresponding dichlorobutadiene ethers, *CH:CCl:CCl:CH(OMe):CH₂*, bo. 103-5.5°, d_4^{20} 1.2278, n_D^{20} 1.4945, and the *Et compd.*, bo. 104-5.5°, d_4^{20} 1.1620, n_D^{20} 1.4924. Hydrolyses of the former by 5% H_2SO_4 at 40-50° gave 50% *Me 1,2-dichlorovinyl ketone*, bo. 61-5.2°, which after bromination and reduction by Zn in AcOH gave $BrMeCO$. **XXII. Order of addition of Br to bromoprene. Synthesis and properties of 1,2-dibromo-1,3-butadiene.** *Ibid.* 237-41(English summary). Bromoprene (12 g.) in 100 cc. $CHCl_3$ was treated at 10-15° with 40 g. Br in 100 cc. $CHCl_3$ to yield 80% *CH₂BrCBr:CH:CH₂Br*, bo. 121-2°, d_4^{20} 2.0852, n_D^{20} 1.6084. Oxidation with $KMnO_4$ yielded only $BrCH_2CO_2H$. The above (15 g.) added to 14 g. KOH in 50 cc. EtOH, let stand 0.5 hr., then steam distd., yielded 6 g. *1,2-dibromo-1,3-butadiene*, bo. 46-6.5°, d_4^{20} 1.9081, n_D^{20} 1.5702, the rest of the material appeared to be the EtO deriv. which were not investigated. The yield of the dibromobutadiene is decreased if the dehalogenation is done at elevated temp. There was evidence of the formation of *bromotoluene* during the dehalogenation. The dibromobutadiene shows polymerization tendency on heating, to yield a viscous vitreous

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CHCX: mono-(III), X = Cl, yields mono-$C_6H_5X_4$ R. C. P.

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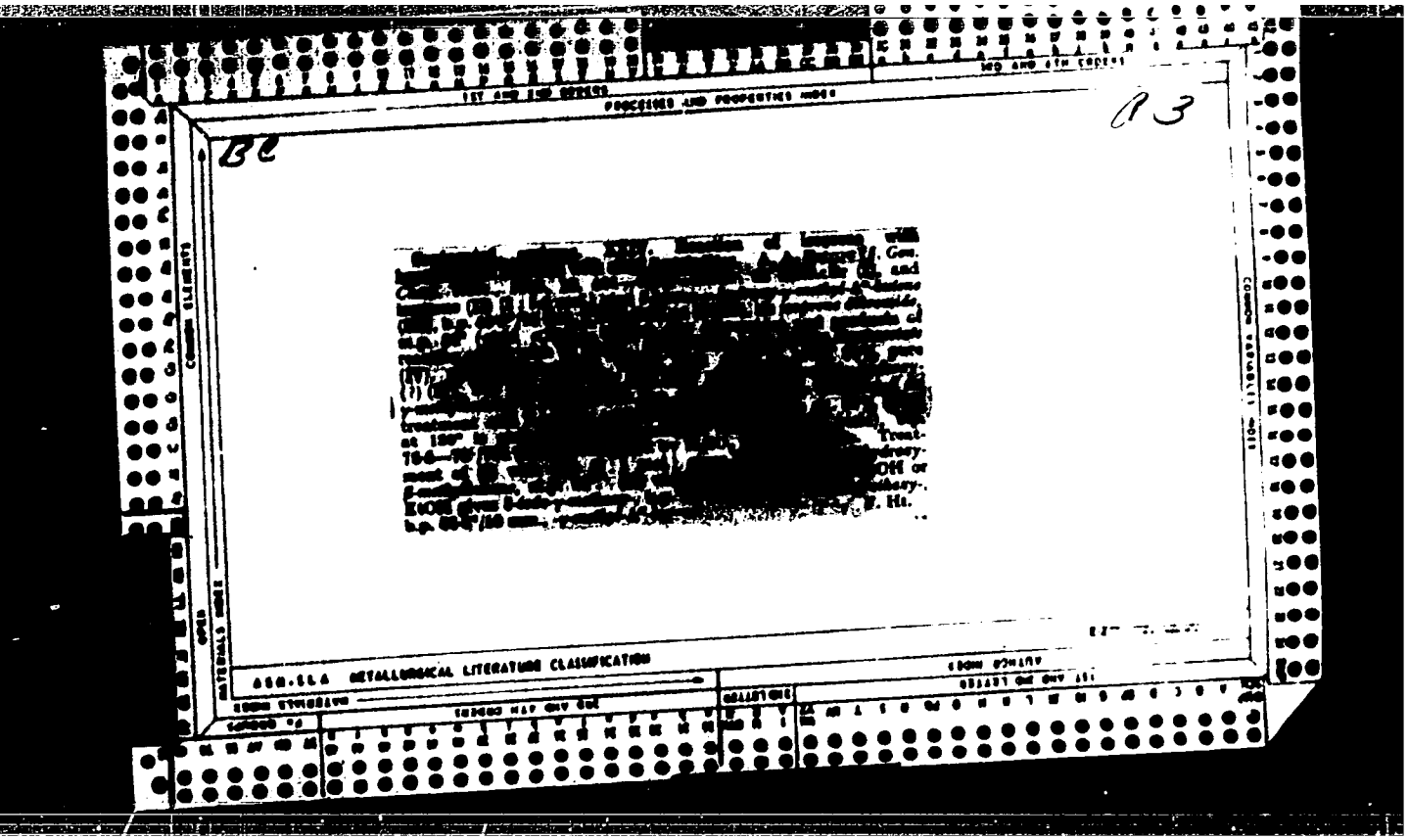

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Conjugated systems. XXV. Isomeric dichlorides and dibromides of isoprene. Synthesis and properties of α- and γ-bromoisoprene. A. A. Petrov. *J. Gen. Chem.* (U. S. S. R.) 13, 741-51(1943)(English summary); cf. *C. A.* 36, 3248[8]. —$Me_2C(OH)CH:CH_2$ (86 g.) in 200 cc $CHCl_3$ at −10 to 5° was treated with Cl until 70 g. wt. gain was obtained; near the end of the reaction evolution of HCl was observed; after washing and distn. the main fraction, $C_5H_8Cl_2$, b. 70.5-1.5° (it is stated that distn. was done *in vacuo*, but the pressure is not indicated), d_4^{20} 1.2208, n_D^{20} 1.4738. Bromination was performed similarly to yield the $C_5H_8Br_2$, b_{10} 94.5°, n_D^{20} 1.5300, d_4^{20} 1.8108. Dehydration with P_2O_5 under reduced pressure at 98° gave the corresponding butenes: *3,4-dichloro-2-methyl-1-butene*, b_{10} 42°, b_{10} 63.5-4.5°, b. 142-3.5°, n_D^{20} 1.4712, d_4^{20} 1.1256, and *3,4-dibromo-2-methyl-1-butene*, b_{10} 46.75°, n_D^{20} 1.5400, d_4^{20} 1.7449, the latter kept at 150° is almost quantitatively transformed into *cis-1,4-dibromo-2-methyl-2-butene*, b_{10} 79-81°, while at higher temp. or longer heating part of the cis compd. is changed into the *trans form*, b_{10} 87-9°. Bromination of isoprene in $CHCl_3$ at −5° yields only *trans-1,4-dibromo-2-methyl-2-butene*, b_{10} 87-9°, n_D^{20} 1.5032, d_4^{20} 1.7000. The dibromide adds a mol. of Br at −5° in $CHCl_3$ to yield apparently a mixt. of the 2 possible isomers, b_{10} 141-2.5°, n_D^{20} 1.6012-1.6030, d_4^{20} 2.3761-2.3819. The 1,4-dibromomethylbutene (11.4 g.) was mixed with 17 g. powd. KOH and heated on a steam bath for 15-20 min., after which the product was distd. *in vacuo* to yield *α-bromoisoprene*, 60%, b_{10} 41-2.5°, n_D^{20} 1.5142, d_4^{20} 1.3271. It forms a *condensate* with maleic anhydride. 3,4-Dichloromethylbutene (7 g.) was reacted with 11.2 g. KOH in 75 cc. ROH in the presence of hydroquinone to yield [illegible] g. dichloroisoprene, b_{10} [illegible]-37°, n_D^{20} 1.4980, d_4^{20} 0.9504, which forms *4-chloro-3-methyl-4-cyclohexene-1,2-dicarboxylic acid*, m. 202-3.5°, with maleic anhydride. By using a similar procedure with 1,4-dibromomethylbutene there was obtained 70% *γ-bromoisoprene*, b_{10} 35°, which yields *4-bromo-3-methyl-4-cyclohexene-1,2-dicarboxylic acid*, m. 201-2°, with maleic anhydride. The same bromoisoprene is obtained from $CH_2CMeC:CH$ by shaking with 50% HBr in the presence of CuBr. 1,4-Dibromomethylbutene in soln. of alkali in alc. yields 75% of *diethers of isoprene glycol, di-Me* b_{10} 44-4.5°, n_D^{20} 1.4334, d_4^{20} 0.9001; *2,3-dibromide*, b_{10} 105-7°, d_4^{20} 1.61; *di-Et*, b_{10} 63-5.1°, n_D^{20} 1.4310, d_4^{20} 0.8885; *1,4-dibromide*, b_{10} 110-21°, d_4^{20} 1.4[illegible]. The reaction of the 3,4-isoprene dichloride or of any of the isoprene dibromides with Zn yields about 65% isoprene. G. M. Kosolapoff

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1ST AND 2ND CODES

3RD AND 4TH CODES

PROCESSES AND PROPERTIES INDEX

Recent advances in the field of halo derivatives of [illegible] diesel/ A A Petrov [illegible] Akimu [illegible] 1944 -A summarizing review of work done in this field by P and other investigators 211 references are given

M Hosch

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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The influence of catalysts on the order of addition of reactants between II and alcohols to propylene oxide

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1ST AND 2ND ORDERS

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3RD AND 4TH ORDERS

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Order of addition of hydrogen halides to organic oxides in aqueous medium. A. A. Petrov (Tashkent Aviation Inst.). *Zh. Obshch. Khim.* (U.S.S.R.) 15, 600-8(1945)(English summary). It was shown that in the reaction of $Me_2C:CH_2$ and $Me_2C:CHMe$ with HOCl, the addn. occurs according to Markownikov's rule, i.e., with predominant formation of tertiary alcs. However, in the addn. of HCl to oxides of the above hydrocarbons, there are formed products which are contrary to the rule, i.e. primary and secondary alcs., resp. Isobutylene (42 g.) was shaken for 1 hr. with a 2% H_2SO_4 soln. of $H_2NCONHCl$, which had been prepd. by adding 30 g. Cl to 45 g. urea in 300 cc. water and 46 g. $CuCO_3$. The reaction mixt. sepd. into 2 layers, the combined aq. layer and water washings of the org. layer were extd. with $(NH_4)_2SO_4$, and extd. with Et_2O to give 65% *isobutylene chlorohydrin*, b_{50} 56.6-7.5°, b_{760} 125.7°, d_4^{20} 1.0502, n_D^{20} 1.4302, which was identified as *dimethyl(chloromethyl)carbinol*. Similarly, $Me_2C:CHMe$ gave 65% of its chlorohydrin, b_{50} 54.5-5°, b_{760} 137.8°, d_4^{20} 1.0465, n_D^{20} 1.4412, which, on the basis of oxidation expts., appeared to be an essentially pure *secondary alc.* Isobutylene oxide (5.2 g.) was added at 5-10° to 15 cc. concd. HCl, and the mixt. was diluted with 4 vols. ice-cold water, in which all but 0.2 g. of the product dissolved readily. The pleasant odor of the insol. portion indicated the presence of dioxolane. After satn. of the aq. soln. with $(NH_4)_2SO_4$ and extn. with Et_2O there was obtained 55% of the *chlorohydrin*, b_{50} 59-61°, b_{760} 126-32° (decompn.), d_4^{20} 1.0472, n_D^{20} 1.4398; this chlorohydrin must be sepd. from the reaction mixt. as rapidly as possible as it tends to split out HCl and form polymers of the resulting aldehyde (which was identified as Me_2CHCHO); small amts. of the corresponding glycol appear to form in the reaction. Oxidation expts. indicated that this chlorohydrin contains about 35% of the tertiary and about 65% of the secondary alc., i.e., the addn. of HCl to the oxide is in reverse order to that of hypochlorination of the olefin. If the addn. of HCl is conducted in dry Et_2O, the results are essentially the same, the resulting chlorohydrin being an approx. equimolar mixt. of the 2 isomers. *Trimethylethylene oxide* (8.6 g.), reacted with 25 cc. concd. HCl as above, yielded 55% of the *chlorohydrin*, b_{50} 55.6°, d_4^{20} 1.0988, n_D^{20} 1.4430, which contained about 30-35% of the secondary alc., as detd. by oxidation expts. Addn. of HCl in dry Et_2O gave a chlorohydrin mixt. which contained 75-80% secondary alc. G. M. Kosolapoff

A10-51A METALLURGICAL LITERATURE CLASSIFICATION

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

The order of addition of hydrogen halides to halogen derivatives of trimethylethylene and isopropylethylene oxides. A. A. Petrov (Tashkent Aviation Inst.) *J. Gen. Chem.* (U.S.S.R.) 15, 931-9(1945) (English summary). The study of addn. of HX to β-halogen deriv. of ethylene oxides provided a rule according to which, predominantly, the addn. takes place with the formation of alcs. with the OH group meso- to the halogen atoms. Cleavage of HX from $Me_2CXCH(OH)CH_2X$ showed that in case of dichlorides or dibromides the tertiary halogen is easiest to cleave, while in the case of chlorobromides, Br is removed first regardless of the position. The halides of trimethylethylene oxide were prepd. by vacuum distn. with KOH of the products of addn. of Cl or Br to $Me_2CH(OH)CH:CH_2$ (cf. *C.A.* 39, 605⁹), the dihalohydrins were added dropwise to a 4-fold excess of 80% KOH under 80-120 mm. to effect the distn. *4-Chloro-2-methyl-2,3-epoxybutane* was prepd. similarly by distn. of $Me_2ClCH(OH)CH_2Cl$ from KOH. The following oxides were prepd. in 75-80% yields: $O:CMe_2CH(CH_2Cl)$, b. 122-3°, d_4^{20} 1.0416, n_D^{20} 1.4338; $Me_2CClCH:CH_2O$, b. 130-1°, d_4^{20} 1.0012, n_D^{20} 1.4370; $O:CMe_2CH(CH_2Br)$, b_{18} 48-25°, d_4^{20} 1.3832, n_D^{20} 1.4642. The oxides were added dropwise at 5-10° to a 4-fold excess of 37% HCl or 50% HBr, after which the mixts. were dild. with water, satd. with $(NH_4)_2SO_4$, extd. with Et_2O, and distd. to yield 80-90% of the dihalohydrins: $Me_2CClCH(OH)CH_2Cl$, b_{20} 79.5-80.5°, d_4^{20} 1.2053, n_D^{20} 1.4732; $Me_2CBrCH(OH)CH_2Cl$, b_{20} 91-2.5°, d_4^{20} 1.4936, n_D^{20} 1.5022; $Me_2CClCH(OH)CH_2Br$, b_{20} 92-4°, d_4^{20} 1.5164, n_D^{20} 1.5012; $Me_2CBrCH(OH)CH_2Br$, b_{20} 100-4°, d_4^{20} 1.7077, n_D^{20} 1.5290. The structure of the alcs. was proved by chromic acid oxidation to the corresponding ketones: $Me_2CClCOCH_2Cl$, b_{20} 72-3°, d_4^{20} 1.2079, n_D^{20} 1.4626; $Me_2CBrCOCH_2Cl$, b_{20} 86-6.5°, d_4^{20} 1.5242, n_D^{20} 1.4935; $Me_2CClCOCH_2Br$, b_{20} 83-3.5°, d_4^{20} 1.5156, n_D^{20} 1.4870; $Me_2CBrCOCH_2Br$, b_{20} 98°, d_4^{20} 1.8110, n_D^{20} 1.5100. Reaction of 3-chloro-3-methyl-1,2-epoxybutane with HBr gave the same chlorobromohydrin as obtained from HCl and 4-bromo-2-methyl-2,3-epoxybutane, b_{20} 92-3°, d_4^{20} 1.5211, n_D^{20} 1.5010. The presence of small amts. of products of "reverse" addn. was shown by dehydration of the crude products by P_2O_5 at 100° and 10-50 mm., followed by treatment of the dihalides with alc. KOH, which gave 10-15% yields (over-all) of *γ-chloroisoprene* and *γ-bromoisoprene*, which were identified by their *maleic anhydride adducts*, m. 203-4° and 202-4°, resp. G. M. K.

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