"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, A.Ye., akademik; SHISHKIN, V.Ye.

Interaction of imidoethers with alkyl halides. Dokl. AN SSSR 141 no.2:349-352 N '61. (MIRA 14:11)

1. Kazanskiy khimiko-tekhnologicheskiy institut im. S.M.Kirova. (Ethers) (Halides)

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ARBUZOV, A.Ye., akademik; SHISHKIN, V.Ye.

Rearrangement mechanism of imido ethers. Dokl. AN SSSR 141 no.3:611-612 N '61. (MIRA 14:11)

1. Kazanskiy khimiko-tekhnologicheskiy institut im. S.M. Kirova.

(Imides) (Ethers)

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, A. YE., VALITOVA, F. G.

Naphtylenesalkyl esters of phosphorus acid.

Khimiya i Primeneniye Fosfororganicheskikh Soyedineniy (Chemistry and application of organophosphorus compounds) A. YE. ARBUZOV, Ed. Publ. by Kazan Affil. Acad. Sci. USSR, Moscow 1962, 632 pp.

Collection of complete papers presented at the 1959 Kazan Conference on Chemistry of Organophosphorus Compounds.

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ARBUZOV, A. YE.

PHASE I BOOK EXPLOITATION SOV/6034

Konferentsiya po khimii i primeneniyu fosfororganicheskikh soyedineniy. 2d, Kazan', 1959.

Khimiya i primeneniye fosfororganicheskikh soyedineniy; trudy (Chemistry and Use of Organophosphorus Compounds; Conference Transactions) Moscow, Izd-vo AN SSSR, 1962. 630 p. Errata slip inserted. 2800 copies printed.

Sponsoring Agency: Akademiya nauk SSSR. Kazanskiy filial.

Resp. Ed.: A. Ye. Arbuzov, Academician; Ed. of Publishing House: L. S. Povarov; Tech. Ed.: S. G. Tikhomirova.

PURPOSE: This collection of conference transactions is intended for chemists, process engineers, physiologists, pharmacists, physicians, veterinarians, and agricultural scientists.

COVERAGE: The transactions include the full texts of most of the scientific papers presented at the Second Conference on the Chemistry and Use of

Card 1/14

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, A.Ye.; VALITOVA, F.G.

Investigations in the field of α,α-diphenyl-β-picrylhydrazine.
Izv. AN SSSR Otd.khim.nauk no.2:354 F '62. (MIRA 15:2)

1. Khimicheskiy institut Kazanskogo filiala AN SSSR.
(Hydrazine)

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, A.Ye., akademik

N.N. Zinin, an outstanding Russian chemist; 150th anniversary of his birth. Vest. AN SSSR 32 no.9:68-77 S '62. (MIRA 15:9)
(Zinin, Nikolai Nikolaevich, 1812-1880)

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, A.Ye.

Great Russian scientist M.V.Lomonosov. Vop.ist.est.i tekh. no.12:3-21 '62. (MIRA 15:4)
(Lomonosov, Mikhail Vasil'evich, 1711-1765)

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, A.Ye., akademik; CHZHAN TSZIN-LIN [Chang Ching-ling]

Interaction of certain substituted triarylchloro- and bromomethanes with salts of dialkylphosphorous acids. Dokl. AN SSSR 144 no.5:1039-1041 Je '62. (MIRA 15:6)

1. Kazanskiy filial Akademii nauk SSR.
(Methane) (Phosphorous acid)

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

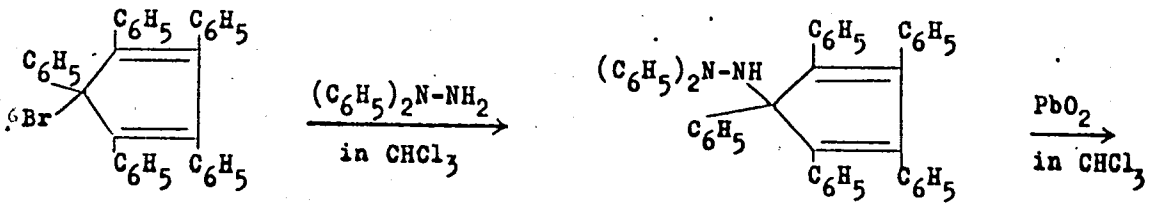
S/020/62/147/001/015/022
B106/B101

AUTHORS: Arbuzov, A. Ye., Academician, Valitova, F. G., Il'yasov, A. V., ~~Kosyrev, B. M.~~, Yablokov, Yu. V.

TITLE: Study of the free radical α,α-diphenyl-β-pentaphenyl-cyclopentadienyl hydrazyl by the e.p.r. method

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 147, no. 1, 1962, 99-102

TEXT: The e.p.r. spectrum of the free radical α,α-diphenyl-β-pentaphenyl-cyclopentadienyl hydrazyl (I) was studied both in solution and in its crystalline state. The synthesis of I was:



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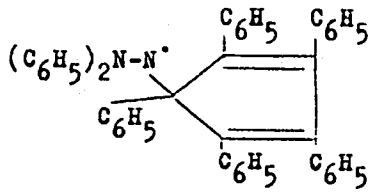
"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5



(I). Data for the radical: yield 70-80%; small bright-orange crystals with a melting point >180°C (decomposition); soluble in benzene, chloroform, alcohol, acetonitrile, glacial acetic acid and dioxane. In dilute solutions (< 10^{-3} moles/l), the spectra show a hyperfine structure, the analysis of which proves that the unpaired electron in I remains mainly on the nitrogen atoms. A comparison of the e.p.r. spectrum of I with the spectrum of the α,α-diphenyl-β-picryl hydrazyl radical (DPPH) showed that the additional hyperfine structure is due solely to the protons of the α-phenyl groups. It may be explained by the interaction of the unpaired electron with the 2,4,6-protons of one of the two α-phenyl groups. The value obtained for the constant a of hyperfine coupling was 1.7 oersteds, and for ΔH_n 1.1 oersteds. The relative

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stability of related free radicals from the e.p.r. spectra are estimated by the method of J. A. Weil, K. V. Sane, J. M. Kinkade (J. Phys. Chem., 65, 710 (1961)) showed that I is chemically more stable than DPPH. Its stability may be due to steric factors reducing the possibility of chemical reactions with other substances. The values obtained from the e.p.r. spectra of I in finely crystalline state, which may contain solvent, were 15.7 ± 0.3 oersteds for ΔH at 295°K, 10.5 ± 0.3 oersteds at 77°K, 1.43 for r at 295°K, and 1.45 at 77°C ($r = \langle \Delta H^4 \rangle^{1/4} / \langle \Delta H^2 \rangle^{1/2}$). The g-tensor at 295°K is: $g_1 = 2.0039 \pm 0.0001$, $g_2 = 2.0051 \pm 0.0001$, and $g_3 < g_1$. The considerable difference between these values and the g-factor of DPPH suggests that the molecular structure of the free radical considerably affects the residual spin - orbital coupling and anisotropy of the g-factor. There are 3 figures and 1 table. The most important English-language references are: M. M. Chen, K. V. Sane et al., J. Phys. Chem., 65, 713 (1961); B. Kubo, K. Tomita, J. Phys. Soc. Japan, 9, 888 (1954); F. K. Kneübuhl, J. Chem. Phys., 33, 1074 (1960).

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Study of the free radical ...

S/020/62/147/001/015/022
B106/B101

ASSOCIATION: Fiziko-tekhnicheskiy institut Kazanskogo filiala Akademii nauk SSSR (Physicotechnical Institute of the Kazan' Branch of the Academy of Sciences USSR); Khimicheskiy institut im. A. Ye. Arbuzova Akademii nauk SSSR (Chemical Institute imeni A. Ye. Arbuzov of the Academy of Sciences USSR)

SUBMITTED: August 8, 1962

Card 4/4

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

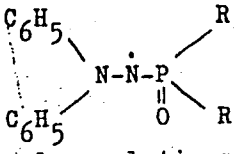
S/020/62/147/004/017/027
B107/B186

AUTHORS: Arbuzov, A. Ye., Academician, Valitova, F. G., Il'yasov, A. V., Kozyrev, B. M., Yablokov, Yu. V.

TITLE: Electron paramagnetic resonance in solutions of some free radicals of the phosphono-hydrazyl series

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 147, no. 4, 1962, 839-842

TEXT: The resonance spectra of the following radicals were studied:



, where R = OC_2H_5 (I), OC_6H_5 (II), C_6H_5 (III). 1.10^{-3} molar solutions in acetonitrile and chloroform were studied. A РЭ -1301 (RE-1301) radiofrequency spectrometer with a 9330 Mc frequency of the magnetic field was used. In all cases, a hyperfine structure of five equidistant lines was caused by interaction of the unpaired electron with the two N^{14} atoms. The spectrum is described by the spin Hamiltonian:

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$\hat{\mathcal{H}} = g\beta H\hat{S} + A_1\hat{S}\hat{I}_{N_1} + A_2\hat{S}\hat{I}_{N_2}$, where β is the Bohr magneton, $g \approx g$ (α,α-diphenyl-β-picryl hydrazyl) = 2.0036, H is the value of the static magnetic field, $S = 1/2$; $I_{N_1} = I_{N_2} = 1$. The constants A_1 and A_2, and the width δH between maximum and minimum of the first derivative of the individual hyperfine structure line were obtained through comparison with theoretically plotted curves, using the given parameters. Calculated data agreed well with those obtained by experiments. $A_1 + A_2$ values found for phosphono-hydrazyls (maximum: 11.4 oe in azetonitrile, minimum: 9.4 in chloroform) were considerably less than the known value of 17.52 oe established for α,α-diphenyl-β-picryl-hydrazyl. A hyperfine structure caused by the P^{31} nucleus was not found. The production of phosphono-hydrazyls followed the reaction

$$(C_6H_5)_2N\text{-}\underset{H}{N}\text{-}P(C_6H_5)_2 \longleftarrow (C_6H_5)_2\text{-}NH_2 \xrightarrow{R_2P(O)Cl} (C_6H_5)_2N\text{-}\underset{H}{N}\text{-}\overset{O}{P}R_2;$$

$(C_6H_5)_2PCl$ and oxidation:

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$$(C_6H_5)_2\underset{\underset{H}{|}}{N}-\underset{\underset{O}{\|}}{N}-PR_2 \xrightarrow{PbO_2} (C_6H_5)_2-N-\dot{N}-\underset{\underset{O}{\|}}{P}R_2.$$

Reaction yields (70-75%) and physical properties of phosphono-hydrazyls were tabulated. There are 1 figure and 2 tables.

ASSOCIATION: Khimicheskiy institut im. A. Ye. Arbuzova Akademii nauk SSSR (Chemical Institute imeni A. Ye. Arbuzov of the Academy of Sciences USSR); Fiziko-tekhnicheskiy institut Kazanskogo filiala Akademii nauk SSSR (Physicotechnical Institute of the Kazan' Branch of the Academy of Sciences)

SUBMITTED: September 15, 1962

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ARBUZOV, A.Ye., akademik

Address at an out-of-town session of the Department of Chemical Sciences of the Academy of Sciences of the U.S.S.R. by Academician A.E.Arbuzov. Izv.Kazan.fil. AN SSSR. Ser.khim.nauk no.6:3-10 '61. (MIRA 16:5)

(Phosphorus-organic compounds)

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, Aleksandr Yerminingel'dovich

[A.M.Butlerov, a great Russian chemist; the centenary of the theory of chemical composition] A.M.Butlerov, velikii russkii khimik; k 100-letiiu teorii khimicheskogo stroeniia. Moskva, Izd-vo AN SSSR, 1961. 42 p. (MIRA 16:8)
(Butlerov, Aleksandr Mikhailovich, 1828-1886)

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, A.Ye.; CHZHAN TSZIN-LIN [Chang Ching-ling]

Interaction of some substituted triaryl halomethanes with dialkyl phosphites. Report No.1: Interaction of p-alkoxy-substituted triphenylhalomethanes with dialkyl phosphites. Izv. AN SSSR. Ser. khim. no.11:1934-1941 N '63.

Interaction of some substituted triaryl halomethanes with dialkyl phosphites. Report No.2: Interaction of halo-substituted triphenyl halomethanes with dialkyl phosphites. Ibid.:1941-1944

Interaction of some substituted triaryl halomethanes with dialkyl phosphites. Report No.3: Interaction of crystalline triphenylchloromethane with sodium diethyl phosphite. Ibid.:1945-1946

(MIRA 17:1)

1. Kazanskiy khimiko-tekhnologicheskiy institut imeni S.M. Kirova.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

KATAYEV, Yu.P.; TROYEPOL'SKAYA, T.V.; ARBUZOV, A. Ye.

Syntheses of heterocyclic compounds based on E. Fisher's reaction. Part 3: Catalysts of an "abnormal" course of reaction. Zhur. ob. Khim. 34 no.6:1835-1843 Je '64. (MIRA 17:7)

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBOUZOV, A. Ye.; SHISHKIN, V. Ye.

Alcoholysis of imidoester haloalkylates. Zhur. ob. khim. 34 no.11: 3579-3582 N '64 (MIRA 18:1)

1. Kazanskiy khimiko-tekhnologicheskiy institut imeni S.M. Kirova.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, A.Ye.; SHAPSHINSKAYA, O.M.

Reactions of exchange decomposition of metallic derivatives of acid amides. Report No.2: Interaction of sodium and silver salts of benzamide with monochloromethyl, monobromomethyl, and monochloromethylethyl ethers. Trudy KKHTI no.30:22-27 '62.
(MIRA 16:10)

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LAVROV, M.I.; NUZHIN, M.T., prof., otv.red.; MARKOV, M.V., prof., red.; DUBYAGO, A.D., prof., red.; ARBUZOV, A.Ye., akademik, red.; NORDEN, A.P., prof., red.; PIS'REV, V.I., prof., red.; TIKHVINSKAYA, Ye.I., prof., red.; FARYSHNIKOV, V.G., dotsent red.; KOLESNIKOVA, Ye. A., dotsent, red.; KOLOBOV, N.V., starshiy prepodavatel', red.; MOROZOV, D.G., dotsent, red.;

[Some statistical regularities of variable stars and their physical interpretation]. Nekotorye statisticheskie zakonomerr ti u zatmennykh peremennykh zvezd i ikh fizicheskoe istolkovar'e. Kazan', 1955. 63 p. (Kazan. Universitet. Astronomicheskaia observatoriia. Biulleten', no. 31) (MIRA 15:10)

1. Rektor Kazanskogo ordena Trudovogo Krasnogo Znameni gosudarstvennogo universiteta im. V.I.Ul'yanova-Lenina (for Nuzhin). 2. Prorektor po nauchnoy rabote Kazanskogo ordena Trudovogo Krasnogo Znameni gosudarstvennogo universiteta im. V.I.Ul'yanova-Lenina (for Markov).

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KUZNETSOV, Vladimir Ivanovich; ARBUZOV, A.Ye., akademik, otv. red.; KATRENKO, D.A., red.

[Advances in the field of catalytic organic synthesis] Razvitie kataliticheskogo organicheskogo sinteza. Moskva, Nauka, 1964. 433 p. (MIRA 17:12)

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

DANILOV, S.N., glav. red.: ARBUZOV, A.Ye., red.; VVEDENSKIY, A.A., red.; VENUS-DANILOVA, E.D., red.; ZAKHAROVA, A.I., red.; IOFFE, I.S., red.; KAVERZNEVA, Ye.D., red.; LUTSENKO, I.F., red.; MISHCHENKO, K.P., red.; NEMTSOV, M.S., red.; PETROV, A.A., red.; FREYDLINA, R.Kh., red.; SHEMYAKIN, M.M., red.; SHUKAREV, S.A., red.; YUR'YEV, Yu.K., red.

[Biologically active compounds] Biologicheski aktivnye soedineniia. Moskva, Nauka, 1965. 305 p.

(MIRA 18:7)

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

DANILOV, S.N., glav. red.; ZAKHAROVA, A.I., red.; ARBUZOV, A.Ye., red.; VVEDENSKIY, A.A., red.; VENUS-DANILOVA, E.D., red.; IOFFE, I.S., red.; KAVERZNEVA, Ye.D., red.; LUTSENKO, I.F., red.; MISHCHENKO, K.P., red.; NEMTSEV, M.S., red.; PETROV, A.A., red.; FREYDLINA, R.Kh., red.; SHEMYAKIN, M.M., red.; SHCHUKAREV, S.A., red.; YUR'YEV, Yu.K., red.

[Problems of organic synthesis] Problemy organicheskogo sinteza. Moskva, Nauka, 1965. 323 p. (MIRA 18:8)

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ARBUZOV, A.Ye.; SHISHKIN, V.Ye.; TYULENEV, S.S.

Imido ethers. Part 1: First haloalkylates of imido thio ethers. Zhur. org. khim. 1 no.8:1442-1444 Ag '65. (MIRA 18:11)

1. Kazanskiy khimiko-tekhnologicheskiy institut imeni Kirova.

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PROCESSES AND PROPERTIES INDEX

22

Composition of turpentine and of turpentine and rosin oils of the factory "Vakhtan." H. Ambuzov. *Zhur. Prikladnoi Khim.* 2, 585-94(1929).—Samples of turpentine, rosin and turpentine oils were sepd. into fractions by vacuum distn. and their phys. consts. detd. On this basis turpentine was found to contain 60-70% of *d*-α-pinene, 15-20% of carenes and 7.4% of a higher-boiling substance which was not detected in turpentine from *Pinus silvestris* (*C. A.* 23, 4057). The turpentine was also different in compn. from the one investigated by Semmler and von Schiller (*C. A.* 22, 241). The rosin oil contained 26% of pinene, 12% of carenes, 42% of menthenols including a component of high optical activity, 30% of high-boiling fractions and residue. Pure terpene hydrate, m. p. 117°, sepd. from high-boiling fractions after standing for a month. The turpentine oil contained 9% of carenes, 40% of menthenols, 25% of a component of high optical activity, 19% of components $b_{10} > 130°$ and 7% of residue. V. K.

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PROCESSES AND PROPERTIES INDEX

Ca

22

Menthenol from turpentine of the factory "Vakhtan." B. Arbuzov. *Zhur. Prikladnoi Khim.* 2, 595-8(1929); cf. preceding abstr.—The characteristics of the purest isolated menthenol fraction were: b_{10} 88.5-89°, b_{760} 213-214°, d_{10} 0.9378, α_D + 16.50°, coeff. of rotatory dispersion (α_V/α_D) 1.147, empirical formula $C_{10}H_{18}O$. The presence of a double bond was established. The oil was fairly fluid, colorless, transparent and possessed a peculiar odor of menthol and mint with a trace of the odor of the mold of tertiary alcs. The characteristics of the component of high optical activity were: b_{10} 98-99°, α_D +43.20°, (α_V/α_D) 1.133. It was a thick, colorless liquid similar to the one described above but with a more pronounced odor of menthol. Its empirical formula was $C_{10}H_{18}O$.

V. Kalichevsky

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

CA

10

Phosphonocarboxylic acids containing an asymmetrical phosphorus atom. A. E. ARBUZOV AND B. A. ARBUZOV. *J. Russ. Phys.-Chem. Soc.* 61, 1599-607(1929).—Earlier attempts to prep. optically active isomerides of phosphinic acid derivatives are briefly summarized, and the prepn. of *phenylphosphonoacetic acid*, HOP(OPh)-CH_2CO_2H and *phenyl-α-phosphonopropionic acids*, HOP(OPh)$CHMeCO_2H$, is described. Phenylphosphonoacetic acid, m. 121.5-2.5°, was obtained as the Et ester, b_7 195-8°, d_4 1.1223, by the action of diisobutyl phenylphosphite, $PPh(OC_4H_9)_2$, b_7 134.5° (from phosphenyl chloride and iso-BuONa), on $BrCH_2CO_2Et$. The alkaloid salts of the acid could not be resolved into optically active components. *Phenyl-α-phosphonopropionic acid*, m. 188-9°, was prepd. by the same method as the Et ester, b_7 191-3°, d_4^{20} 1.10335, from diisobutyl phenylphosphite and $MeCHBrCO_2Et$.

B. C. A.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

BC

a-3

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

CA

22

Composition of turpentines. B. A. Arbuzov. *Zhur. Prikladnoi Khim.* 3, 867-73 (1930); cf. *C. A.* 23, 4057; 24, 1209. —A sample of com. turpentine (d_{15} 0.866), α_D^{15} + 22.53°, coeff. of rotary dispersion (α_v/α_j) 1.136 after treatment with lime and distn. with steam) was distd. in the app. previously described (*C. A.* 24, 3896) and the fractions were analyzed. It contained *d*-α-pinene 58.3, *d*-Δ^3-carene 20.5, dipentene and *d*-limonene 10.6, terpinolene with traces of α-terpinene 3.0, alcs. 6.0, sesquiterpenes 1.0 and residue 1.5. V. Kalichevsky

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Sesquiterpenes from the turpentines of Pinus sylvestris. B. A. ARBUZOV. *J. Russ. Phys.-Chem. Soc.* 62, 2023-6(1930).—A new *sesquiterpene* isolated from turpentine b_{5} 126.5-8.5°, d^{13} 0.933, $[\alpha]_D$ —30.45° (*dioxide*, b_{5-8} 134-6°, d_0^{20} 1.0175). B. C. A.

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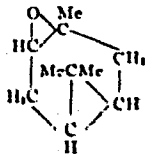
Oxide of *d*-Δ^3-carene. B. A. Arbuzov and B. M. Mikhailov. *J. Russ. Phys.-Chem. Soc.* **62**, 607-15(1930).—*d*-Δ^3-*Carene* (I) belongs to the group of bicyclic terpenes, widely distributed in nature, which was discovered by Simonsen (*C. A.* **14**, 2617). Δ^3-Carene and Δ^4-carene, which was found by S. in the essential oil of *Andropogon Jawarancusa* (*C. A.* **17**, 91), are the first natural terpenes having the carene nucleus:

$Me_2C.CH.CH.CH_2.CMe.CH.CH_2$ (I) and $Me_2C.CH.CH.CH.CMe.CH_2.CH_2$.

S. (l. c.) oxidized I with $KMnO_4$ in alk. soln. to a carenoglycol, m. 69-70°, $[\alpha]_D$ 16.05°, and with H_2O_2 in AcOH to a glycol m. 90-1°, optically inactive in alc. and $CHCl_3$ (Pillay and S., *C. A.* **22**, 1980). This isomerism of glycols was presumed by him to be either a *cis-trans* or optical isomerism, wherefore the glycol m. 69-70° was named *d*-carene-α-glycol, and the glycol m. 90-1° *d*-carene-β-glycol. By the action of dil. H_2SO_4 the β-glycol loses 1 mol. of H_2O with formation of a mixt. of *p*-cymene and

1-epoxy *d*-Δ^3-carene



(II), which has an odor resembling linalool

and proved to be a very stable compd. giving no reaction with Br in $CHCl_3$ and forming no hydrate on shaking for several days with dil. H_2SO_4. The uncommon stability of II as contrasted with the high reactivity of the oxides of α-pinene and norpinene seems to be little understood, and is here proposed to be investigated. The Δ^3-carene isolated

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from turpentine gives on oxidation with BzO_2H (Prilezhaev, *C. A.* **24**, 607) 70% of the oxide of *d*-Δ^3-carene (III), which differs sharply from II prepd. by S., *i. e.*, II (fraction 1) b_{10} 150-3°, d_{20}^{20} 0.961, II (fraction 2) b_{10} 155-9°, d_{20}^{20} 0.9794, $[\alpha]_D^{20}$ −39.10; III b_{10} 79-80°, b_{100} 126-7°, d_{20}^{20} 0.9464, $[\alpha]_D^{20}$ 13.05 $\alpha\nu/\alpha\gamma$ 1.124; III is a light liquid with a strong pine oil odor, and gives readily with dil H_2SO_4 a hydrate, the main fraction of which is cryst. The *d*-carene-β glycol of S. m. 89-90°, optically inactive in $CHCl_3$. The formation of β-glycol by hydration of III indicates for the α- and β glycols of *d*-Δ^3-carene *cis-trans*, and not optical, isomerism, whereby the lower melting α-glycol is the *cis*-, while the β-glycol is the *trans*-isomer (Nametkin, *C. A.* **20**, 2820; Verkade, *et al.*, *C. A.* **23**, 2095). The formation of identical glycols by hydration of an oxide and by oxidation of the corresponding unsatd. hydrocarbon with H_2O_2 in AcOH, as in the case of I, was previously observed (Sword, *C. A.* **19**, 2942); Prilezhaev, *C. A.* **4**, 916, Meerwein, *C. A.* **20**, 2674). *Exptl. part.*—From 4200 g. of Russian turpentine of unknown compn. was obtained by distn. *in vacuo* 845 g. (20.1%) of carene fractions, from which was isolated the pure *d*-Δ^3-carene, b_{10} 57-8°, b_{100} 171-2.5°, d_{20} 0.8568, $\alpha\nu$ 14.30°, $\alpha\gamma$ 16.55°, $\alpha\nu/\alpha\gamma$ 1.157. The nitrosate of I was prepd. by known methods (*C. A.* **23**, 4057). After recrystn. from a mixt. of $CHCl_3$ and MeOH it decomps. 145°. Δ^3-Carene-α-glycol was prepd. by methods developed by Simonsen (*loc. cit.*), Semmler and Schiller (*C. A.* **22**, 241), and Krestinskii and Solodkii (*C. A.* **23**, 4564), m. 70-1°. Δ^3-Carene-β-glycol was prepd. according to Simonsen (*C. A.* **22**, 1969), m. 89.5-90.5°. The oxide of I was obtained by pouring with stirring an ice-cold soln. of 87 g. of I in 300 cc. of anhyd. Et_2O into an ice-cold soln. of BzO_2H in 1200 cc. of Et_2O contg. 10.3 g. of active O; after 26 hrs. of standing the reaction mass was washed 2 times with alkali, dried with fused K_2CO_3, the Et_2O expelled and the residue (96 g.) fractionated at 12.5 mm. pressure, which finally produced the fraction b_{10} 79-80°, or b_{100} 126-7°, yield 53.7 g. or 70%. For the hydration of the oxide of Δ^3-carene, 5 g. of the latter were shaken 1 hr. at room temp. with 25 cc. of 1% H_2SO_4, the ppt. of the glycol was filtered off, washed with H_2O and dried in a vacuum desiccator over H_2SO_4; recrystd., it m. 89-90°; yield 50%. The work is being continued.

CHAS. BLANC

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B-II-9

Composition of the resin of *Pinus insignis*, Douglas. B. Abramov, V. Abramov, and F. Valitova (J. Gen. Chem. Russ., 1932, 2, 376—387).—The resin consists of about ½ turpentine, ½ cryst. resin acids, and ½ amorphous, non-volatile material. The turpentine contains α- and β-pinene with 0·3% of camphene, isolated as isoborneol. The resin acid, $C_{20}H_{30}O_2$, m.p. 143—145°, $[\alpha]_D$ −89·2° in EtOH, α_0/α_1 1·173, resembles α-aleppic and sondarsapinic acid (A., 1929, 573) (NH_3 salt, m.p. 55°); it is rapidly isomerised to abietic acid by HCl in EtOH, the lavorotation of the solution rapidly decreasing at first and then increasing. One double linking in the acid is almost instantly oxidised by BzO_2H or AcO_2H, the second very slowly. G. A. R. K.

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Co

22

...benzene ring

Turpentine from Pinus pithyusa. B. A. Arbuzov, L. Bastanova, E. Ivanova, L. Kozlovskaya, A. Maltzeva and V. Fedotov. *J. Applied Chem.* (U. S. S. R.) 5, 787-9(1932).—Analytical results are given. V. Kalichevsky

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Presence of conjugated bonds in abietic acid. B. Arbuzov. *J. Gen. Chem.* (U. S. S. R.) 2, 806-13(1932).—While the presence of 2 double bonds in abietic acid (I) was conclusively demonstrated, their position was not made clear. The 2 double bonds show different reactivity giving easily the dihydro and difficultly the tetrahydro deriv. of I, and showing unlike speed in the oxidation with BzO_2H. From the formula of I proposed by Ruzicka (R. and Pfeiffer, *C. A.* 20, 421; R. *et al.*, *C. A.* 25, 3657) there could be assumed the presence of conjugated bonds, an assumption here investigated by the action of maleic anhydride (II) on I. I, m. 166°, $[\alpha]_D$ −78.9° (alc.), obtained from rosin acids by isomerization with HCl, was heated 4 hrs. at 170° in a sealed tube with II in dry C_6H_6, extd. with Et_2O and crystd. by adding petr. ether to incipient turbidity, giving an addn. compd., $C_{28}H_{38}O_5$ (III), crystals from 100% AcOH, m. 227°. III, treated with NaOH and then acidified, gave the corresponding acid, which on heating was reconverted without melting to the anhydride, m. 227°. III (4 g.) with 6 g. powd. Se, heated 24 hrs. at 280-320°, then extd. with Et_2O and distd. *in vacuo*, produced 60% retene, crystals from MeCN, m. 97-8°, which with CrO_3 in AcOH gave retenequinone, m. 196-7°. The formation of retene does not indicate the position of the double bonds in I, and could be thus explained: (1) at the temp. of dehydrogenation (300-20°) III is decompd. into II and I, which is then converted to retene; (2) the cleavage of the new cycle takes place, because of its position in the mol. of III; (3) the newly formed ring generally can not remain during the dehydrogenation. The 1st case is excluded, because III heated 30 min. at 300-20° in a metal bath split off no II, the reaction beginning at 385-400° with the formation of $(CH_3CO)_2O$, m. 118.5-9.5°. The other 2 cases are closely related and will require addnl. material for their interpretation. III (1.5 g.), treated with the calcd. amt. of aq. NaOH and then with 0.8 g. $KMnO_4$ in 120 cc. H_2O, satd. with CO_2, evapd., acidified, filtered, washed and dried over H_2SO_4, produced 1.2 g. $C_{28}H_{38}O_5$, crystals from H_2O, m. 191-2°, which is presumed to be the monolactone, and not the anhydride, of the expected dihydroxytricarboxylic acid. It was interesting to investigate whether the

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ability of I to give III depends on the method of prepn. of I, because some authors presume that the acids obtained from resin do not contain double bonds, but decomp. with the rupture of the ring and the formation of double bonds in the process of production by the action of HCl or vacuum distn. (Tyutyunnikov and Perstnev, C. A. 26, 3395). I, obtained by vacuum distn. of various rosins and the primary resinous acids heated with II, produced in 5 expts. III, m. 227°, identical with III described above, while I, m. 173–4°, $[\alpha]_D$ −19.81°, obtained from French rosin by vacuum distn., and I, m. 158–61°, $[\alpha]_D$ ± 0°, from the rosin of *Pinus silvestris* heated 24 hrs. at 270° with II, gave no addn. products. The exptl. data conclusively prove the presence of conjugated double bonds in I. The work is being continued.

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Isomerization of α-pinene to an aliphatic terpene. I, II. Examination of the aliphatic terpene. B. A. Arbuzov, *J. Gen. Chem.* (U. S. S. R.) 3, 21-7, 28-34(1933).—When α-pinene (I) is passed over Cu chromite (Adkins, *C. A.* 25, 1797, 1808) at 300°, some 80% of I is recovered and 20% of an aliphatic terpene II. At 375°, 23% of I, 42% of dipentene and 31% of II are produced, and at 400-10°, 20% of I, 23% of dipentene, 20% of II and 30% of an unidentified monocyclic terpene $C_{10}H_{16}$. Dipentene is not isomerized under these conditions. II may be identical with the product obtained from I with an Al catalyst. A Co-Th catalyst at 380° gives similar products in comparable yield (cf. *C. A.* 26, 2582). II, b_{16} 87-7.5°, d_4^{20} 0.8102, n_D 1.5448, $[\alpha]_D$ 0, closely resembles allo-ocimene; it is readily oxidized in the air to a viscous product with the properties of a peroxide. II is reduced by Na and EtOH to a *dihydro compd.*, b. 168.5-70°, contg. 2 double linkings (titration with AcO_2H). Reduction of II over a Cu-Cr catalyst gives 2,6-dimethyloctene, b. 165-6°, and H_2 and Ni give *dl*-2,6-dimethyloctane, b. 159.5°. II combines with maleic anhydride to an *anhydride*, $C_{14}H_{18}O_3$ (III), m. 81-2°, hydrated to an *acid*, $C_{14}H_{20}O_4$, m. 154.5-5.5°, which is isomerized by HBr to an *acid*, m. 189-90°, dehydrated by Se to a compd., m. 77.5-80° (IIIP). II and citraconic anhydride give an *anhydride*, $C_{13}H_{18}O_3$, m. 82°. B. C. A.

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CA

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Dihydroterpene. B. A. Arbuzov. Russ. 30,100, Mar 31, 1934. Dihydroterpene is prepd. by passing α-pinene at elevated temp. over a catalyst consisting of oxides of Cu, Cr, Co, Th or their mixts. and reducing the product with Na in an alc. medium.

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Composition of Russian turpentine. B. A. Arbuzov. *J. Applied Chem.* (U. S. S. R.) 7, 757-9; *Trans. Butlerov Inst. Chem. Tech. Kazan* No. 1, 48-50(1934).—Careful fractionation of large amts. of turpentine from *Pinus silvestris* by the Damois-Dupont method gave *d*-α-pinene 76.48, *d*-Δ^3-carene 13.67, *l*-terpene 0.87, higher fractions 1.20 and losses 1.78%. A. A. Bochtlingk

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Diene synthesis. B. A. Arbuzov, E. P. Salmina and O. M. Shaimkinskaya. *Trans. Bashkir Inst. Chem. Tech. Kazan*, No. 2, 9-18(1934).—No condensation products were formed on heating maleic anhydride (I), m. 53-5°, b. 202°, in sealed glass tubes in the presence of dry xylene 3 with α-vinylnaphthalene (II) at 100° for 6 hrs. and with Ph_2 at 170° for 4.5 hrs. and at 200° and 210-15° for 3-3.5 hrs., and β-quinone with Ph_2 at 200° for 3 hrs. Unlike I, citraconic anhydride did not react with $Ph_2C{:}CH_2$ on heating the mixt. at 150° for 10 hrs. Thus, in these cases the double bond of the PhH ring does not form a conjugated bond with the side chain, and according to Wagner-Jauregg (*C. A.* 26, 982) I can combine in other than the 1,4-position. In the production of II by the method of 4 Tiffeneau and Dandel (*C. A.* 3, 810) were obtained II and a new compd. α-$C_{10}H_7CH(OH)Me$, m. 64°, b_{10-6} 170-5° with a partial cleavage of H_2O. Chas. Blanc

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B-II-8

Composition of Russian turpentine oil. B. A. [illegible] (J. Appl. Chem. Russ., 1934, 7, 757—759).—The product (from Pinus silvestris) contains d-n-pinene [illegible], d-Δ³-[illegible] 13·87, l-terpene 6·87, and higher-b.p. fraction 1·20%. R. T.

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The structure of abietic acid. II. The synthesis of 1,3-dimethylcyclohexane-1,2,3-tricarboxylic acid. B. A. Arbuzov and O. M. Shapshinskaya. *Trans. Kiev. Inst. Chem. Tech. Kazan* No. 3, 19-34(1935); *Ber.* 68B, 437-42 (1935); cf. *C. A.* 27, 2088.—By oxidation of abietic acid (I) with $KMnO_4$ Ruzicka *et al.* (*C. A.* 29, 421) obtained an acid (II), $C_{11}H_{16}O_6$, m. 218-9° (tri-Me ester, m. 75°), presumably 1,6-dimethylcyclohexane-1,2,3-tricarboxylic acid (*C. A.* 25, 3657). The work of Vocke (*C. A.* 26, 4950) and Haworth, *et al.* (*C. A.* 26, 4337) indicated the structure *1,3-dimethylcyclohexane-1,2,3-dicarboxylic acid*. Ruzicka later (*C. A.* 27, 2154) suggested a formula for I based on the latter structure of II. A. and S. now report an attempt to synthesize II. Condensation of trimethylene bromide with 2 mols. of $NaCMe(CO_2Et)_2$ followed by hydrolysis gave α,α'-dimethylpimelic acid (III), a mixt. of the para and anti forms. As by-products in the condensation were formed a colorless liquid of unknown compn., b_{10} 100-2, n_D^{20} 1.4335, d_4^{20} 1.0067, $C_{10}H_{18}O_4$, and a colorless liquid, $C_{11}H_{20}O_6$, b_4 137-9°, n_D^{20} 1.4350, d_4^{20} 1.0000, mol. refraction 67.07, probably *di-Et methylethoxypropylmalonic ester* (IV). IV hydrolyzed to an acid, b_4 175-8°, not obtained pure. III was converted to *di-Et α,α'-dimethyl-α,α'-dibromopimelate* (V), colorless liquid, $b_{4.4}$ 182.5-4°, n_D^{20} 1.4888, d_4^{20} 1.4284, mol. refraction 81.19. V with $Na_2C(CO_2Et)_2$ gave *tetra-Et 1,3-dimethylcyclohexane-1,2,2,3-tetracarboxylate* (VI), b_4 208-8°, d_4^{20} 1.1054, n_D^{20} 1.4613, mol. refraction 90.26. With VI was also produced *di-Et 1,5-dimethylpenta-1,4-diene-1,5-dicarboxylate* (VII), b_4 138-9°, $n_D^{20.5}$ 1.4775, d_4^{20} 1.0451, mol. refraction 64.94. Hydrolysis of VII gave 2 stereoisomeric *1,5-dimethylpenta-1,4-diene-1,5-dicarboxylic acids*, m. 165-8° and m. 191-3°. VI on hydrolysis gave the corresponding acid which rapidly lost CO_2. Elimination of CO_2 in *vacuo* at 100-5° gave an acid which could not be cryst. and hence identification with II was not possible. Purification through the Pb and Ba salts did not succeed. The Ag salt reacted with MeI to give a tri-Me ester which boiled similarly to that from II but could not be cryst. The analyses of the tri-Me ester and the Ag salt corresponded to II.

Lewis W. Butz

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Isomerization of α-pinene to an aliphatic terpene (allocimene). VI. Isomerization of nopinene, Δ-Δ²-carene, camphene, and pinocamphone. B. A. Arbuzov (Trans. Kirov Inst. Chem. Tech. Kazan, 1955, No. 4—5, 53—56; cf. A., 1935, 1246).—allOcimine is obtained in 20% yield by passing nopinene over glass at 245—350°; under these conditions Δ²-carene, camphene, and pinocamphone do not undergo conversion into (I). R. T.

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3RD AND 4TH ORDERS

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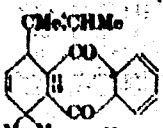
a-3

Isomerisation of α-pinene into the aliphatic terpene allocimene. III. Conditions of formation and properties of the terpene. IV. Structure of the terpene. B. A. ARBUSÓV (J. Gen. Chem. Russ., 1936, 6, 206—216, 217—226).—III. *allo*-Ocimene (I) is obtained by passing α-pinene over reduced CuO (26·5% yield) or glass (24·6% yield), at 340—350°. (I) in EtOH and naphthaquinone (100°; 3 hr.) yield the substance (II), m.p. 122°, from which 2 H are eliminated (C atoms 11 and 12) by atm. O_2 in presence of KOH, to afford a *product*, m.p. 129—130·5°. This is oxidised by HNO_3 (200°; 5 hr.) to yield a *substance*, m.p. 331°, probably a naphthaquinonedicarboxylic acid. (I) in AcOH and H_2SO_4 afford dipentene, a diterpene, b.p. 142—143°/4 mm., and polyterpenes (not isolated).



(II.)

IV. $CMe_2:CH\cdot CH_2Br$ and $CHMe:CMe\cdot CHO$ in Et_2O are added to Mg, when an alcohol, $C_{10}H_{18}O$, b.p. 82—83°, is obtained. This is dehydrated by $KHSO_4$ at 180° to yield an unidentified *terpene*, b.p. 160·5°, not identical with the expected (I), and condensing with maleic anhydride to afford a *product*, m.p. 70—71°. (I) obtained from pinene is shown to be identical with natural (I), and with that obtained synthetically by the method of Fischer and Löwenberg (A., 1933, 592).

R. T.

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ARBOUZOW, B.A.

"Sur l'isomerisation de l' -pinene en terpene aliphatique (alloocimene). Communication V". Arbouzow, B.A. (p. 292)

SO: Journal of General Chemistry. (Zhurnal Obshchei Khimii) 1936, Vol. 6, No. 2

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Ca

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Isomerization of α-pinene to an aliphatic terpene (allo-ocimene). VI. Isomerization of nopinene, *d*-Δ³-carene, camphene and pinocamphone. B. A. Arbuzov. *J. Gen. Chem.* (U.S.S.R.) 6, 297-9(1936); cf. *C. A.* 29, 1407⁸.—Nopinene was partially isomerized to alloocimene by passing its vapors over fragments of Suprex glass at 345-50°. *d*-Δ³-Carene, camphene and pinocamphone failed to isomerize under these conditions and in the presence of reduced Cu at 300-400°. Chas. Blanc

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d-3

Structure of abietic acid. II. Attempted synthesis of 1 : 3-dimethylcyclohexane-1 : 2 : 3-tricarboxylic acid. B. A. ARBUSOV and O. M. SCHAPSCHINSKAJA (J. Gen. Chem. Russ., 1936, 6, 404—416).—$CH_2Br\cdot CH_2\cdot CH_2Br$ and $CNaMe(CO_2Et)_2$ in EtOH (at the b.p.) yield the Et_4 ester of αα-dimethylpentane-αααα-tetracarboxylic acid (I), together with by-products, amongst which $OEt\cdot CH_2\cdot CH_2\cdot CH_2\cdot CMe(CO_2Et)_2$ is identified. (I) and aq. EtOH–KOH (5 hr. at the b.p.) afford αα-dimethylpimelic acid, which, with red P and Br (2 hr. at 100°), followed by EtOH (3·5 hr. at the b.p.), yields Et_2 αα-*dibromo*-αα-*dimethylpimelate*, b.p. 182·5—184°/2·4 mm., and this is condensed with $CNa_2(CO_2Et)_2$ in EtOH (100°; 3 hr.) to give the Et_4 ester of 1 : 3-*dimethylcyclohexane*-1 : 2 : 2 : 3-*tetracarboxylic acid* (II), b.p. 206—208°/2 mm., together with some Et_2 αα-*dimethyl*-$\Delta^{\alpha\beta}$-*pentadiene*-αα-*dicarboxylate*, b.p. 138—139°/2 mm., from which the *acid* $CH_2(CH{:}CMe\cdot CO_2H)_2$, m.p. 168—171°, is obtained by hydrolysis. The acid obtained by hydrolysis of (II) (EtOH–KOH) yields a glassy mass, $C_{11}H_{16}O_5$ (Me_2 ester, b.p. 158—162°/0·7 mm.), when heated in vac. at 160—165°. The structure of the final product was not definitely established. R. T.

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3RD AND 4TH ORDERS

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a-3

Isomerisation of oxides of terpenes. I. Isomerisation of α-pinene oxide by Reformatski's reaction. B. A. Arbusov (J. Gen. Chem. Russ., 1936, 6, 417—422).—α-Pinene oxide (I), Zn, and $CH_2Br\cdot CO_2Et$ in C_6H_6 (2 hr. at the b.p.) afford the substance $\begin{matrix}CHMe\cdot CMe_2\\ OH\text{——}CH_2\end{matrix}\!\!>\!CH\cdot CH(OH)\cdot CH_2\cdot CO_2Et$ (II), b.p. 146—147°/4 mm., from which the corresponding acid, m.p. 74—75°, is obtained by hydrolysis (MeOH–NaOH). (I) and $ZnBr_2$ in C_6H_6 (at the b.p.) afford campholenaldehyde, which yields (II) when treated with Zn and $CH_2Br\cdot CO_2Et$. R. T.

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Ca

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The reaction of dienes with diazo compounds. II. Arbuzov and S. Ruthov. *J. Gen. Chem.* (U. S. S. R.) 17, 2195-2201 (1947); cf. *C. A.* 39, 3072⁸.—In the reaction of dienes with diazotized p-nitroaniline (I), some dinitrodiazoaminobenzene is always formed. Hence, the method of Tereat'ev, Vinogradov and Hal'pern (*C. A.* 30, 3746⁶) for detg. dienes is unreliable. If I is diazotized in strong HCl-AcOH with excess $NaNO_2$, and the excess of the latter destroyed with urea, bivinyl will couple with the diazo compd. The product, which m. 118-19°, is probably $O_2NC_6H_4N{:}NCH{:}CHCH{:}CH_2$. When it is reduced with $SnCl_2$ and HCl, it forms p-$C_6H_4(NH_2)_2$ (II) and pyrroline. This shows that coupling occurs in the α-position of the diene. Ring closure may occur during coupling, but more probably takes place during the reduction. I and 2,4-hexadiene give an analogous compd., m. 172-3°. Its reduction with Zn dust and HCl gives II and 2,5-dimethylpyrroline. H. M. Leicester

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B. ARBUZOV

Some products of the diene synthesis with piperylene and butadiene. B. Arbuzov, F. Zimovets and I. Plotnikova. *J. Gen. Chem.* (U. S. S. R.) 7, 2278-91(1937).—The hexadiene fraction of the divinyl still residues from synthetic rubber manuf. condenses with acrolein to give 2,5-dimethyl-3-cyclohexen-1-al (I) and with crotonaldehyde to give 2,5,6-trimethyl-3-cyclohexen-1-al (II). The piperylene fraction and acrolein give 5-methyl-3-cyclohexen-1-al (III). I condenses with MeEtCO to form a ketone which is probably RCH:CHCOEt, but may be RCH:CMeCOMe (R = 2,5-dimethyl-3-cyclohexen-1-yl). It b_{10} 132-5°, d_4^{20} 0.9275, n_D^{20} 1.4930, M. R. 60.15. With MePrCO, I gives an analogous compd. which is either RCH:CHCOPr or RCH:CHCOMe, b_{10} 154-5°, d_4^{20} 0.9143, n_D^{20} 1.4921, M. R. 65.30. I and mesityl oxide give RCH:CHCOCH:CMe_2, b_9 138-40°, d_4^{20} 0.9121, n_D^{20} 1.4960, M. R. 70.00. III and MeEtCO give R'CH:CHCOEt or R'CH:CMeCOMe, b_{10} 140°, d_4^{20} 0.9225, n_D^{20} 1.4883, M. R. 55.62 (R' = 5-methyl-3-cyclohexen-1-yl). I and EtCHO give RCH:CHCH$_2$CHO, b_{10} 120-1°, d_4^{20} 0.9055, n_D^{20} 1.4976, M. R. 51.01. III and EtCHO give R'CH:CHCH$_2$CHO, b_{10} 110-13°, d_4^{20} 0.9415, n_D^{20} 1.4910, M. R. 49.42, and mono-α-methyl-β-ethylacrolein. III condenses with EtOAc and Na to give R'CH:CHCO$_2$Et, b_{10} 124-5°, d_4^{20} 0.9628, n_D^{20} 1.4820, M. R. 57.44. The Et acetal of I, b_{10} 117-19°, d_4^{20} 0.9200, n_D^{20} 1.4570, M. R. 62.85; the Bu acetal, b_9 153-5°, d_4^{20} 0.9003, n_D^{20} 1.4611, M. R. 81.57. The Et acetal of III, b_{10} 91-3°, d_4^{20} 0.9260, n_D^{20} 1.4544, M. R. 57.95. By the Grignard reaction with I the following alcs. are prepd.: RCH(OH)Et (IV), b_{10} 120-3°, d_4^{20} 0.9340, n_D^{20} 1.4825, M. R. 51.29; RCH(OH)Pr, b_{10} 120-2°, d_4^{20} 0.9200, n_D^{20} 1.4880, M. R. 56.01; iso-BuCH(OH)R, b_{10} 124-6°, d_4^{20} 0.9378, n_D^{20} 1.4860, M. R. 59.87; RCH(OH)Ph, b_{10} 167-8°, d_4^{20} 1.0000, n_D^{20} 1.5493, M. R. 66.71. Oxidation of IV with $K_2Cr_2O_7$ gives the corresponding ketone, b_{10} 100-2°, d_4^{20} 0.9272, n_D^{20} 1.4700; semicarbazone, m. 100°. II gives by the Grignard reaction R'CH(OH)Et (R' = 2,5,6-trimethyl-3-cyclohexen-1-yl), b_{10} 116-8°, d_4^{20} 0.9240, n_D^{20} 1.4820, M. R. 56.16; R'CH(OH)Pr, b_{10} 118-20°, d_4^{20} 0.9112, n_D^{20} 1.4822, M. R. 61.35 and iso-BuCH(OH)R', b_{10} 132-4°, d_4^{20} 0.9142, n_D^{20} 1.4841 M. R. 65.76. All the compds. except the aldehydes derived from condensation with EtCHO have pleasant odors

H. M. Leicester

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BC

cyclo-Dehydration of certain substituted camphenilanols. B. A. ARBUSOV (J. Gen. Chem. Russ., 1939, 9, 339—348).—Camphenilanealdehyde (I) and $CH_2Ph \cdot MgCl$ in Et_2O yield *benzylcamphenilanylcarbinol*, b.p. 183—185°/5 mm., m.p. 71—72°, which with 90% H_2SO_4 at >20° affords 1 : 1-*dimethyl*-2 : 12-*endomethylene*-1 : 2 : 3 : 4 : 9 : 10 : 11 : 12-*octahydrophenanthrene*, b.p. 132·5—133·5°/1 mm. *Phenylcamphenilanylcarbinol*, b.p. 153—155°/3 mm., m.p. 80—81°, similarly yields a *hydrocarbon*, $C_{18}H_{22}$, b.p. 110·5—111°/0·3 mm., which may be 1 : 1-dimethyl-2 : 11-*endomethylene*-1 : 2 : 3 : 4 : 10 : 11-hexahydrofluorene, or 9 : 9-dimethyl-1 : 6-*endomethylene*-2 : 3-benzo-1 : 3 : 3-di-*cyclo*-Δ^8-nonene. (I) and $1\text{-}C_{10}H_7 \cdot MgCl$ afford unstable β-naphthylcamphenilanylcarbinol, which splits off H_2O during distillation, yielding 2-α-*naphthylidene*-1 : 1-*dimethyl*-3 : 6-*endomethylenocyclohexane*, b.p. 185·6—188°/1 mm. (*picrate*, m.p. 111—112°); this undergoes condensation in presence of H_2SO_4, giving a *hydrocarbon*, $C_{18}H_{22}$, b.p. 185—186°/1 mm., probably 8:8-dimethyl-9:12-*endomethylene*-7:8:9:10:11:12:13-heptahydrobenzanthrene. The product of condensation of campholenealdehyde and $1\text{-}C_{10}H_7 \cdot MgCl$ reacts with H_2SO_4 yielding a *hydrocarbon*, $C_{18}H_{22}$, b.p. 165—167°/0·5 mm., probably a cholane derivative.

R. T.

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Isomerization of terpene oxides. II. Isomerization of α-pinene oxide in the Grignard reaction. B. A. Arbuzov. *J. Gen. Chem.* (U. S. S. R.) 9, 249–54(1939); cf. *C. A.* 29, 6887°.—The results of an independent study on the Prilezhaev and Yershuk reaction of condensation of α-pinene oxide (I) with the Grignard reagent (cf. *C. A.* 23, 4463) agree with the findings of Ritter and Russel (*C. A.* 30, 2948°) that the product of reaction is an alkyl- or aryl-substituted campholenol and not $CH_2:CH:CR.CH(OH)$.-$CH_2.CHCHMe_2$ (II) as was shown by P. and V. The reaction of 40 g. Et_2Zn and 50 g. *l*-I at 160° and subsequent hydrolysis with H_2O and soln. in HCl proceeds without the isomerization and rupture of the pinane nucleus to give 18 g. of an *alc.*, $C_{10}H_{18}O$, b_{14} 81.5–2.5°, $d_4^{20.5}$ 0.9248, n_D^{20} 1.475, M. R.$_D$ 45.42, $[\alpha]_D$ 10.04° (cf. Cook, *et al.*, *C. A.* 30, 2502°). I with PhLi failed to react. III. **Isomerization of the oxides of campbene, nopinene and Δ^3-carene in the Reformatsky reaction.** *Ibid.* 255–71.—Studies on the isomerization of α-pinene oxide with the intermediate formation of campholenaldehyde (I) in the Reformatsky reaction are extended to addnl. bicyclic terpenes. Camphene oxide (II), nopinene oxide (III) and *d*-Δ^3-carene oxide (IV) were prepd. from the corresponding terpenes by oxidation with AcO_2H in Et_2O. Refluxing 25 g. II, m. 86.8°, $[\alpha]_D$ 16.62, in 50 ml. of dry C_6H_6 with 0.2–0.3 g. $ZnBr_2$, removal of the C_6H_6 and redistn. of the residue afforded 20 g. I. II gave as sole product with Zn and $BrCH_2CO_2Et$ (V) an *ester*, $C_{12}H_{20}O_3$ (VI), b_{10} 131.5–2.5°, d_4^{20} 1.0403, n_D^{20} 1.483, M. R.$_D$ 65.32, saponif. to a *acid*, m. 111–12°. Since VI also resulted by the action of Zn and V on I, it appears to be the *Et ester of β-hydroxy-δ-12,2-dimethylbicyclo[1.2.2]heptyl-3)propionic acid*. III, b_{14} 68.5–9.5°, d_4^{20} 0.9693, n_D^{20} 1.478, M. R.$_D$ 44.36, does not react with $ZnCl_2$ and $ZnBr_2$ in Et_2O, but in C_6H_6 it is isomerized into *dihydromyrtenal*, b_{10} 81–2°, d_4^{20} 0.9023, n_D^{20} 1.4812, M. R.$_D$ 44.96; *semicarbazone*, m. 152–3°. III with Zn and V gave a *said. ester* (VII), $C_{12}H_{20}O_3$, b_{14} 137.8°, d_4^{20} 1.0278, n_D^{20} 1.48, M. R.$_D$ 60.33. It appears to be the *Et ester of β-hydroxy-δ-(7,7-dimethylbicyclo[1.1.3]heptyl-2-)propionic acid*. IV, b_{10} 80–80.5°, d_4^{20} 0.9578, n_D^{20} 1.460, M. R.$_D$ 44.24, $[\alpha]_D$ 11.95. The complex reaction of IV

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ARBUZOV, B.A.; ABRAMOV, V.S.; DEVYATOV, YA. B.

"The Products of Condensation of Cyclones with p-Benzoquinone and Napthoquinone", Zhur. Obshch. Khim., 9, No. 17, 1939. Laboratory of Organic Chemistry, Kazan' Chemico-Technological Institute imeni S. M. Kirov. Rec'd 22 Feb 1939.

[redacted] Report U-1614, 3 Jan 1952.

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ARBUZOV, B. A.

"Uber Die Struktur Der -Pimarsaure," (About the structure of Primary Acids), Iz. Ak. Nauk USSR, Ot. Khim. Nauk, No. 1, 1940

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Modern views regarding the composition of the non-volatile constituents of the resins of conifers and the structure of the resin acids. II. Atbusov. *Lesokhim. Prom.* 3, No. 5, 3-8(1940); *Chem. Zentr.* 1941, I, 580.—Recent investigations indicate that the so-called sapinic acids contained in the conifer resin are only a mixt. of a few primary resin acids and their conversion products (secondary resin acids). It is possible that they consist only of the *d*- and *l*-primary acids and the pro- and dihydroabietic acids. A summary is given of the structure, properties and phys.-chem. consts. of these acids and of the abietic and the pyroabietic acids. The literature on the subject is also reviewed.

M. G. Moore

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3RD AND 4TH ORDERS

Ca

10

The action of hexadiene and piperylene upon *p*-quinone. B. A. Arbuzov and S. M. Spekterman. *Trans. Kirov. Inst. Chem. Tech. Kazan* No. 8, 21-5(1940).—The condensation of 75% hexadiene (8 g.) with 8 g. *p*-quinone in 8 cc. of $PhNO_2$ in a sealed glass tube at 130-40° for 6 hrs. yielded 35% of *1,4-dimethyl-1,4,4a,8a-tetrahydronaphthoquinone*, $\begin{matrix} CH.CHMe.CH.CO.CH \\ \| \quad\quad\quad\quad \| \\ CH.CHMe.CH.CO.CH \end{matrix}$, m. 187-8°. No pos. results were obtained in attempts to add a 2nd mol. of hexadiene to the above product. Oxidation of the product with CrO_3 in a glacial AcOH while cooling with water yielded *1,4-dimethylnaphthoquinone*, m. 124-5°. The reaction between hexadiene and *p*-quinone in pyridine and $PhNO_2$ mixt. at 120-5° for 12 hrs. in a sealed tube yielded *1,4-dimethyl-1,4-dihydronaphthoquinone*, m. 179-80°. The condensation of 30 and 84% piperylene fractions with *p*-quinone in abs. alc.-$PhNO_2$ mixt., and $PhNO_2$ under similar conditions as above, yielded *1-methyl-1,4,4a,8a-tetrahydronaphthoquinone*, m. 159-60°. A. A. P.

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CA

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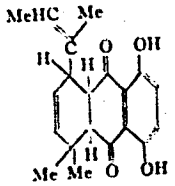
Action of naphthazarin on hexadiene and piperylene. B. A. Arbuzov and K. Nikanorov. *J. Gen. Chem.* (U. S. S. R.) 10, 649-52(1940).—Condensation of hexadiene (I) and piperylene (II), resp., with naphthazarin (III) was carried out first in abs. alc. with the formation of resins. When $PhNO_2$ was used instead of alc., and the mixt. was heated in a sealed tube at 100-70° for 2 hrs. *1,4-dimethyl-5,8-dihydroxyanthraquinone* was obtained from I and III, orange-red needles, m. 220-7°, in 73.7% yield, and *1-methyl-5,8-dihydroxyanthraquinone* from II and III, when heated in a sealed tube at 125-30° for 30 hrs., red needles, m. 230-7°, yield 42.7% (based on III).



(V)

When benzene was used as reaction medium, negative results were obtained. Condensation of III with allo-ocimene (IV) in abs. alc. in CO_2 atm. in a sealed tube at 80-90° gave a reaction product $C_{26}H_{32}O_4$, red needles, m. 157°, yield 20.5%. The same compd. was obtained when benzene was used, whereas no cryst. product could be isolated in the case of $PhNO_2$. Though the structure for this compd. has not yet been proved, formula V is suggested because of results obtained in previous studies on the condensation of α-naphthoquinone with IV.

Gertrude Berend

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ARBUZOV, B. A.

"On the Structure of ℓ-Pimaric Acid," Dok. AN, 30, No. 8, 1941. State Univ. Kazan. Butlerov Chem. Research Inst.

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ARBUZOV, B. A., Butlerov, A. M.

"Structure of l-pimaric Acid," Organic Chem Lab, Inst of Chem Research Investigation, Kazan State Univ., Uchenye Zapiski Kazan Gosudarst Univ, Vol. 101, No. 1, 1941

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ARBUSOW, B. A.

"The action of dihydronaphtalenes on cyclones." Arbusow, B. A., and Akhmed-Zade, J. A. (p. 211)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii) 1942, Vol 12, No 3-4.

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ARBUSOW, B. A.

"Piperylcyclone and its derivatives." Arbusow, B. A., and Akhmed-Zade, J. A. (p. 218)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii) 1942, Vol 12, No 3-4.

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a-3

Derivatives of l-pimaric acid. [illegible] Chem. Russ., 1942, 12, 342–[illegible]. [illegible] is given of some reactions and [illegible] of l-pimaric acid obtained during the [illegible] structure of this primary pine resin acid [illegible]

l-Pimaric acid (I) [illegible] with 1:2-$O_2C_6H_4SO$ (1·9 g. of [illegible]) [illegible] 9 g. of crude [illegible] to give a compound [illegible] which effloresces in air and [illegible] derivative, m.p. 176–177° [illegible] in MeOH. The [illegible] H_2SO_4 at [illegible] and other products of [illegible] constitution. G. S. S.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, Boris Aleksandrovich

"Use of Parachors and Dipole Moments for Studying the Fine Structure of Some Organic Derivatives of Phosphorous, Boron, Nitrogen, and Silicon." Zhur. Obsch. Khim., 13, Nos. 1-2, 1943

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Addition products of dienes to toluene. B. A. Arbuzov and E. V. Kurnetsov. *Compt. rend. acad. sci. U.R.S.S.* 39, 311-13(1943)(in English).—Toluene (I) and butadiene (II) were caused to react in the presence of *finely divided* Na and the products were cyclized and then dehydrogenated with S. Reaction of 300 g. I and 200 cc. II in presence of Na dust at 90° for 10 hrs. yielded 142 g. of a product (b_{10} 80-220°) whose fractionation yielded *1-phenyl-2-pentene* (III), b_{10} 92-4°, n^{20}_D 1.5090, d^{20}_4 0.876; *9-phenyl-2,6-nonadiene* (IV), b_{10} 140-2°, n^{20}_D 1.5105, d^{20}_4 0.889; $PhCH_2(C_4H_6)_2H$, b_{10} 188-91°, n^{20}_D 1.5125, d^{20}_4 0.898; $PhCH_2(C_4H_6)_3H$ b_{10} 210-20°, n^{20}_D 1.5135, d^{20}_4 0.920. Cyclization of III yielded 95% of 1,2,3,4-tetrahydro-1-methylnaphthalene (V), which with S yielded 75% of dehydrogenation product, b_{10} 145°, n^{20}_D 1.6130, d^{20}_4 1.001. Similarly IV produced 90% of cyclization product, b_{10} 162-4°, n^{20}_D 1.5471, d^{20}_4 0.980, thought to be *1-ethylhexahydrobenzonaphthene*, whose dehydrogenation yielded no pos. result. Condensation of V with II at 160° with Na catalyst yielded 15% of a product (b_{10} 140-2°, n^{20}_D 1.5378, d^{20}_4 0.953) formulated as *1-(2-butenyl)-1,2,3,4-tetrahydronaphthalene*, which was cyclized to a product b_{10} 148-50°, n^{20}_D 1.5548, d^{20}_4 1.0008, which resinified on attempted dehydrogenation with S. Conjugated dienes with substituents on the terminal C atoms reacted much less readily with I than did II. Thus 300 g. I and 250 g. 2,4-hexadiene with 20 g. Na at 70° for 10 hrs. yielded only 40 g. of a product from which was isolated *1-phenyl-2-methyl-3-hexene*, b_{10} 100-10°, n^{20}_D 1.4905, d^{20}_4 0.875. J. W. Perry

The m-monohalogenated benzotrifluorides. G. C. Finger and M. L. Kanowski. *Trans. Illinois State Acad. Sci.* 37, 66-8(1944).—*m*-Bromobenzotrifluoride (b. 151-3°) and *m*-iodobenzotrifluoride (b_{20} 82-2.5°) were prepd. from m-$H_2NC_6H_4CF_3$ by the Sandmeyer reaction with yields of 44% and 81%, resp. The introduction of a halogen always raises the b.p. of a compd., the increment being less as we pass down the series from F to I. The ds. are also higher and the indexes of refraction are lower in comparison with the toluene analogs or the simple halobenzenes. J. H. Reedy

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

Ca

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Use of dihalo ethers for the synthesis of oxygen-containing cyclic acids. I. Ali-Zade and E. A. Arbuzov. *J. Gen. Chem.* (U. S. S. R.) 13, 113-23(in English, 123-4) (1943).—Contrary to the data of Dox and Yoder (*C. A.* 16, 1562), the authors showed that the reaction of $(BrCH_2)_2O$ with metallic derivs. of malonic ester yields the normal product, $[(HO_2C)_2CHCH_2]_2O$. Mg (40 g.) and 100 cc. abs. EtOH were treated with 250 g. $CH_2(CO_2Et)_2$ (after addn. of 1/6 of the malonate the mixt. was heated on a steam bath for 45 min. and 1 cc. CCl_4 added to initiate the reaction). After complete soln. of the Mg, the flask was cooled and 150 g. $(BrCH_2)_2O$ was added dropwise, after which the mixt. was heated on a steam bath for 2 hrs., cooled, treated with dil. H_2SO_4, extd. with Et_2O, the latter dried and distd., yielding 100 g. of a heavy oil, b_6 168-71°, d_4^{20} 1.1128, n_D^{20} 1.4382, and 26 g. of material b_6 171-205°, which crystallizes on standing and m. 50-2° (from aq. EtOH); the former compd. is *bis(2,2-dicarbethoxyethyl) ether*, while the 2nd substance was not investigated further. Hydrolysis of the ester by KOH in aq. EtOH gave a *tetrabasic acid*, m. 147-9°. The ester (20 g.), 2.7 g. Mg and 20 cc. EtOH were heated on a steam bath and treated with 0.5 cc. CCl_4 to initiate the reaction, after which the heating was continued until the Mg disappeared; on cooling the mixt. was treated with 10 g. iodine in EtOH and heated on a steam bath for 8 hrs.; after the usual treatment with acidified water and extn. with Et_2O, there was obtained 8 g. *tetra-Et tetrahydro-3,3,6,6-furanstetracarboxylate*, b_6 171-4°, d_4^{20} 1.1293, n_D^{20} 1.4461. Similar reaction with CH_2Br_2 gave *tetra-Et tetrahydro-3,3,5,5-pyrantetracarboxylate*, b_6 179-82°, d_4^{20} 1.1131, n_D^{20} 1.4390, while $(CH_2Br)_2$ gave *tetra-Et hexamethylenoxide-3,3,6,6-tetracarboxylate*, $b_{1.4}$ 158-60°, d_4^{20} 1.1110, n_D^{20} 1.4385; $Cl(CH_2)_2Br$ gave $(EtO_2C)_2CHCH_2OCH_2C(CO_2Et)_2CH_2CH_2CH_2Cl$, b_1 102-6°, d_4^{20} 1.1026, n_D^{20} 1.4395; $(:CHCH_2Br)_2$ gave 35% of $O.CH_2.C(CO_2Et)_2.CH_2.CH:CH.CH_2.C$-

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$(CO_2Et)_2.CH_2$ (I), b_1 101-4°, d_{21}^{21} 1.1000, n_D^{20} 1.4519; $(BrCH_2)_2O$ gave 45% of $O.CH_2.C(CO_2Et)_2.CH_2.O.CH_2.C(CO_2Et)_2.CH_2$ (II), b_5 185-9°, d_{21}^{21} 1.1415, n_D^{21} 1.4530.

$(BrCH_2CH_2)_2O$ was prepd. by treating a cooled mixt. of 12 g. dry pyridine and 44 g. $O(CH_2CH_2OH)_2$ with 88 g. PBr_3; after standing overnight the product was distd. in vacuo, washed, dried and redistd., b_{20} 110°. Na (2.3 g.) in 50 cc. EtOH was treated with 15 g. of *tetra-Et 1,1,2,2-ethanetetracarboxylate* in 25 cc. EtOH; after heating on a steam bath for 1 hr., the mixt. was treated with 8 g. $(BrCH_2CH_2)_2O$ and heated on a steam bath for 7 hrs., the EtOH distd. off, the residue decomposed with water and extd. with Et_2O; on distn. of the ext. there was obtained 8 g. *tetra-Et hexamethylenoxide-4,4,5,5-tetracarboxylate*, b_5 208-12°, d_{21}^{21} 1.1572, n_D^{21} 1.4561. $[CH_2CH(CO_2Et)_2]_2$ (from $(CH_2Br)_2$ and $CH_2(CO_2Et)_2$) in the form of its Mg salt was treated with $(BrCH_2CH_2)_2O$, yielding *tetra-Et octamethylenoxide-4,4,7,7-tetracarboxylate*, in 17% yield, $b_{1.5}$ 190-4°, m. 75-7°. The Na compd. from 19.2 g. Na, 300 cc. EtOH and 80 g. $CO(CH_2CO_2Et)_2$ was treated with 80 g. $(BrCH_2CH_2)_2O$, heated for 18 hrs., the NaBr sepd., the soln. heated for 62 addnl. hrs. with several sepns. of NaBr; the EtOH was then distd., the residue treated with water and extd. with Et_2O, yielding 34 g., b_4 158-63, d_{21}^{21} 1.1562, n_D^{21} 1.4615, of an oil which corresponded to $CH_2.CH_2.O.CH_2.CH_2.O.C(CH_2CO_2Et):CCO_2Et$. Hydrolysis of this substance with alc. KOH gave a monobasic acid, m. 87-9°, $C_5H_{12}O_4$, which appears to be either $CH_2.CH_2.O.CH_2.CH_2.O.C(CH_2CO_2H):CH$ or $CH_2.CH_2.O.CH_2.CH_2.O.CMe:CCO_2H$.

G. M. K.

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ARBUZOV, B. A., Rafikov, S. R., and Zoroastrova, V. M.

"Preparation of the Dinitrile of Adipic Acid" Inst Org Chem, Acad Sci, Moscow, Bul Acad Sci URSS, Class Sci Chim, 1945

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3RD AND 4TH ORDERS

DC

AP

Diene compounds. I. Simultaneous action of chlorine and ethylene oxide on butadiene. B. A. Arbusov and V. M. Zoroastrova (*Bull. Acad. Sci. U.R.S.S., Cl. Sci. Chim.*, 1945, 113—119).—Contrary to Petrov's conclusion that hypohalous acids and their esters add to $CH_2CH{-}CHCH_2$ only in the 1 : 2-positions, addition of $Cl{-}CH_2CH_2{-}OH$ takes place in the 1 : 4-positions. Cl_2 and $(CH_2{-}CH)_2O$ are passed simultaneously into $CH_2{-}CH{-}CH{-}CH_2$ (100 g.), initially at −30° to −35°, until the wt. of reaction mixture increases to 300 g. After 6 hr. at 5—8°. The products isolated are: $CH_2Cl{-}CHCl{-}CH{-}CH_2$ (20 g.); $CH_2Cl{-}CHCH{-}CH_2Cl$ (19·6 g.), α-chloro-β-β′-chlorohydrin-but-γ-ene (II) (80·7 g.), b.p. 72—75°/8 mm., α-chloro-β-β′-chlorohydrin-but-δ-ene (III) (64·6 g.), b.p. 105°/10 mm., a fraction (19·0 g.), b.p. 120—130°/10 mm.; not yet fully investigated, but thought to be $(CH{-}CH_2)_2O{-}CH{-}CH{-}CH_2(Cl)_2$, and a residue (98·0 g.). The above structures are inferred from the conversion of (II), by KOH in EtOH, into β-β′-chlorohydrin-α-γ-diene, b.p. 87—88°/10 mm., which polymerises when kept and is hydrolysed by aq. H_2SO_4 to $CO{-}CH{-}CH{-}CH_2$, and from the hydrolysis of (III), by KOH in EtOH, to α-chloro-β-γ-chlorohydrin-β-ene, b.p. 111—112·5°/13 mm., which with Br in $CHCl_3$ forms a *dibromide*, b.p. 163—164·5°/11 mm. G. W.

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ARBUZOV, B. A.

"On the Preparation of Dinitrile of Adipic Acid," Iz. Ak. Nauk SSSR, Otdel. Khim. Nauk, No. 2, 1945. Inst. of Org. Chem, Acad Sci, USSR.

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Dienic compounds. I. Simultaneous action of chlorine and ethylene oxide on butadiene. B. A. Arbusov and V. M. Zoroastrova. *Bull. acad. sci. U.R.S.S., Classe sci. chim.* 1945, No. 2, 113-18(in English, 118-19).—The reaction of chlorine and ethylene oxide on butadiene has been studied. Besides the chlorination of butadiene, the addn. of the elements of $ClCH_2CH_2OCl$ at the 1,2- and 1,4-positions, and the addn. of 2 mols. of $ClCH_2CH_2OCl$ take place. All the products of addn. have been isolated.

G. Lebedeff

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ARBUZOV, B. A., Zelinskiy, N. D. and Shuikin, N. I.

"Oxidation of Cyclohexene by Selenious Acid and a New Method of Preparation of 1, 3-cyclohexadiene," Bul. Acad. Sci. URSS, Classe sci. chim. 1945, 163-6.

SO; Chemical Abstracts, Vol. 40, No. 12, 20 Jun 46

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ARBUZOV, B. A.

The isopropyl ester of benzenephosphonous acid. ~~B. A. Arbuzov~~, G. Kh. Kamai, and O. N. Belorossova (Kazan Chem.-Tech. Inst.). *J. Gen. Chem.* (U.S.S.R.) 15, 766-9 (1945).—When iso-PrOH and $PhNMe_2$ in dry Et_2O are cooled and treated with $PhPCl_2$ in a CO_2 atm., they form a distillate, 60% of which is *di-iso-Pr benzenephosphonite* (I), b_{10} 121-2°, d_4^{20} 1.0100, d_4^{40} 0.9952, n_D^{20} 1.5021, and 15% *iso-Pr phenylisopropylphosphinate* (II), b_{11} 146-7°, d_4^{20} 1.0957, d_4^{40} 1.0813, n_D^{20} 1.4929, formed by isomerization of I. When I is heated to 150° in the presence of iso-PrI (III) it liberates $MeCH:CH_2$ and forms *phenylisopropylphosphinic acid*, m. 61-2°, which gives cryst. Na, K, Ca, and Ba salts. When I and III are allowed to stand 10 days at room temp., 11.1% isomerization to II occurs. Addn. of a little $PhNMe_2$ increases the isomerization to 95%.

H. M. Leicester

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BC

a-9

[illegible] V. S. [illegible] and L. A. [illegible] Sri. U.R.S.S. [illegible] of tetraphenylcyclo- [illegible] n-C_9H_{19} [illegible] (tube) in [illegible] to occur; the 2 : 5-H_2 derivatives are [illegible] 20 hr.), [illegible] 330°; [illegible] tetrahydronaphthalene (147°; 4 hr.), [illegible] 16 hr.), [illegible] (175—184°; 24 hr.) give [illegible] of crude 2 : 5-H_2 derivative; the best yields of recryst. product (m.p. [illegible] or 70% from n-C_9H_{19} or EtOH, respectively. [illegible] also gives an unknown product of high m.p. [illegible] tetrahydronaphthalene affords 70—80% of crude 2 : 5-H_2 compound; e.g., tetrahydronaphthalene at 140—150° (20 hr.) or 150—200° (6 hr.) gives 30% of recryst. product.

A. T. P.

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Reduction of cyclones by hydrocarbons. B. A. Arbuzov, V. S. Abramov, and L. A. Shapshinskaya. *Doklady Akad. Nauk S.S.S.R.* 40, 162-4(1945); *Compt. rend. acad. sci. U.R.S.S.* 46, 147-9(1945)(in English).—Tetraarylcyclopentadienones ("cyclones"; cf. *C.A.* 29, 7323[1]) were partially hydrogenated to the corresponding dihydro derivs. by heating (180-330°) with various inert solvents (xylene, toluene, benzene, tetralin, hexane, EtOH) in sealed tubes in the absence of a catalyst. The identity of the dihydro derivs. so obtained was proved by mixed m.pts. with prepns. prepd. by other synthetic methods (cf. *C.A.* 30, 7349[9]). When EtOH was the H donor, AcH was detected in the reaction mixt.

J. W. Perry

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ARBUZOV, B. A.

Reduction of nitriles of dibasic acids over Raney nickel. B. A. Arbuzov and E. A. Fazhil'tsova. *Bull. acad. sci. U.R.S.S., Classe sci. chim.* 1946, 65-70(in Russian).—The dinitriles of adipic, sebacic, and succinic acids are readily reduced over Raney Ni at 75-80° to the corresponding amino nitriles under 0.5-0.8 atm. H; adiponitrile is reduced to $(CH_2)_6(NH_2)_2$ at 100-10° and 80-100 atm. H. The catalyst was made from Al_2Ni alloy by treatment with boiling 25% NaOH; after decanting, a fresh batch of alkali was used and the mixt. heated 1 hr. at 90°; this was repeated 2-3 times and the catalyst was washed free of alkali by H_2O and abs. EtOH and was used immediately. Adiponitrile (10 g.), 150 g. BuOH, and 5 g. catalyst were treated with H (0.5-0.8 atm.) at 75-80° 10 hrs.; after filtration and removal of the BuOH *in vacuo*, the residue was dild. with H_2O and 0.5 g. unreacted dinitrile was removed. Treatment of the residue with BzCl resulted in isolation of 53% *Bz deriv.* of the resulting *ε-aminocapronitrile*, m. 92-3° (from 70% alc.); hydrolysis by HCl gave *ε-aminocaproic acid-HCl*, which gave the *free acid*, m. 200-2°; the same yield was obtained if the catalyst was added in portions during the course of reduction. Succinonitrile (8 g.), 150 cc. BuOH, and 4 g. catalyst similarly treated with H 19 hrs. at 75-80° gave the *Bz deriv.* of *γ-aminobutyronitrile* (no data given, but a general statement indicated a yield of 90%), as an oil, which on hydrolysis by HCl gave *γ-aminobutyric acid*, m. 181-2°; *HCl salt*, m. 134-5°; *chloroplatinate*, m. 218-20°. Sebaconitrile (10 g.), 150 g. BuOH, and 5 g. catalyst were hydrogenated as above for 26 hrs.; filtration, removal of the BuOH, and treatment of the residue with BzCl gave (yield not given) the *Bz deriv.* of *ε-aminocaproic acid*, m. 103-5°, which on hydrolysis by HCl gave the *free acid*, m. 185-7°; *chloroplatinate*, m. 298-302° (decompn.). Adiponitrile (50 g.), 450 g. BuOH, and 17 g. catalyst heated 2 hrs. to 110° at 90 atm. initial H pressure (45 atm. final) gave 1.3 g. unreacted dinitrile, and 46 g. (85.7%) *hexamethylenediamine*, b. 200-4°; 3.5 g. of tar was formed. Increase of the temp. to 150° (14 hrs.) dropped the yield to 26.8%; halving of the catalyst amt. dropped the yield to 64%. The high-pressure runs were made in a rotating Bergius autoclave.

G. M. Kosolapoff

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ARBUZOV, B. A.

Condensation products of dimethyloladipamide and dimethylol derivatives of amides of other dibasic acids with aromatic compounds. B. A. Arbuzov and D. A. Livshits. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1946, 391-402.—Adipamide (25 g.), heated on a water bath to 70-80° with 42.5 g. 30% formalin, 25 ml. H_2O, and 2.5 g. K_2CO_3 until soln. took place, then an addnl. 2 hrs., and cooled, gave 71% *N,N'-dimethyloladipamide*, $(CH_2CH_2CONHCH_2OH)_2$ (I), m. 148-9.7° (from H_2O). To 8.5 g. C_6H_6 and 42 ml. concd. H_2SO_4 was added gradually 10 g. I at 8-10° and after 3 days at room temp. the mixt. was poured on ice, and the resulting solid washed with H_2O and alc., boiled with H_2O, and extd. with hot alc., giving an amorphous insol. portion, m. 225-7°, and a sol. portion, m. 174-5°, the total yield being 92%. The sol. product, $C_{20}H_{24}O_2N_2$, gives $BzOH$ on oxidation with $KMnO_4$, and appears to be *$PhCH_2NHCO(CH_2)_4CONHCH_2Ph$*. The insol. product, $C_{14}H_{18}O_2N_2$, gives terephthalic acid on oxidation by $KMnO_4$ and appears to be either a polymer —*$(C_6H_4CH_2NHCO(CH_2)_4CONHCH_2)_n$*— or a cyclic product *$p$-$C_6H_4(CH_2NHCOCH_2CH_2)_2$*. AcNHPh (2.6 g.) in 20 ml. concd. H_2SO_4 treated at 0° with 2 g. I, allowed to stand 2 days, poured on ice, and the resulting solid washed with water and recrystd. from AcOH, yielded 1.7 g. (41%) $C_{18}H_{20}O_4N_2$, m. 244-4.5°, which on boiling with 20% HCl gave *p-$H_2NC_6H_4CH_2NH_3$.HCl*, decomp. above 300°; the product is apparently *p-AcNHC$_6$H$_4$CH$_2$NHCO(CH$_2$)$_4$CONHCH$_2$C$_6$H$_4$NHAc*, as on boiling with 30% NaOH it gives 88% *p-$H_2NC_6H_4CH_2NHCO(CH_2)_4CONHCH_2C_6H_4NH_2$*, m. 173-4° (from EtOH). Coupling of the diazotate of this diamine with $PhNMe_2$ in HCl gave the corresponding *[p-$Me_2NC_6H_4N$:$NC_6H_4CH_2NHCO(CH_2)_4$]$_2$*, m. 166-70°; *2HCl*, black-green, m. 185-6° (from AcOH); similar coupling with 2-naphthol in the presence of Na_2CO_3-NaOH gave an analogous *2-hydroxy-1-naphthyl deriv.*, m. 244-6° (by pptn. from H_2SO_4 by H_2O). Condensation of *p-acetotoluidide* with I in H_2SO_4, as above, gave 55% *$C_{20}H_{24}N_4O_4$*, m. 233-4° (from AcOH), which probably has a structure similar to that of the AcNHPh deriv., while *o-acetotoluidide* gave a similar *product*, m. 245-6° (from EtOH). Condensation of I with *2-acetonaphthalide* in H_2SO_4 at 12-15° gave a similar *product*, m. above 300°, insol. in the usual org. solvents. Addn. of 2 g. I to cold 2.7 g. *p-$O_2NC_6H_4OH$* in 20 ml. concd. H_2SO_4, followed by 24 hrs.' standing, gave 80% *[5,2-(NO_2)(OH)C_6H_3-$CH_2NHCO(CH_2)_4$]$_2$*, m. 239-40°, yellow, sol. in alkalies, AcOH, and EtOH; hydrolysis by concd. HCl gave *2-hydroxy-5-nitrobenzylamine-HCl*, m. 240° (from H_2O). 2-Naphthol (2.8 g.) and 2 g. I in 20 ml. EtOH and 20 ml. H_2O treated with 40 drops of concd. H_2SO_4 gave after standing 3 days 50% colorless *[2-$HOC_{10}H_6CH_2NHCO$-$(CH_2)_4$]$_2$*, m. 222-4° (from AcOH), which on hydrolysis

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Allylic rearrangements. I. Isomerization of some methoxychloropentenes in the presence of metal chlorides. A. N. Pudovik and B. A. Arbuzov. *Izvest. Akad. Nauk S.S.S.R., Otdel Khim. Nauk* 1946, 427-37 (in Russian); cf. *C.A.* 42, 4973b.—The addn. of $ClCH_2OMe$ to butadiene was improved over the procedure given by Strauss and Thiel (*C.A.* 31, 3619). Butadiene (230 g.) cooled to −15° was treated with 310 g. $ClCH_2OMe$, followed by 4-5 g. powd. $ZnCl_2$, the flask closed with a stopper provided with an outlet tube and pinch-clamp, and the mixt. carefully mixed by shaking and allowed to stand in an ice-bath 5-6 hrs., then 15-20 hrs. at room temp. When the pressure drops the reaction is complete; the residual butadiene is removed and the residue, dild. with Et_2O, is washed with H_2O and distd. to give 135 g. *1-methoxy-3-chloro-4-pentene*, b_{11} 34-5°, d_4^{20} 0.9990, n_D^{20} 1.4364, 105 g. *1-methoxy-5-chloro-3-pentene*, b_{11} 58.9°, d_4^{20} 1.0000, n_D^{20} 1.4540, and 75 g. high-boiling products. The addn. does not go without the catalyst, even after 1 yr. standing. Neither the 3- (I), nor the 5-Cl isomer (II) shows isomerization on standing 1 yr. at room temp. in sealed tubes. When I and II were heated to 100° in sealed tubes, I isomerized to the extent of 16% in 27 hrs., while II gave but 3% isomerization, as detd. by n. If the temp. was raised to 140° the reaction was speeded up, but incipient decompn. limited the duration to 15 hrs., at which point I was 65.9% isomerized and II 7.9%. The isomerization was also studied in the presence of catalysts: 1% and 10% $ZnCl_2$, and 10% $SnCl_4$. $ZnCl_2$ (10%) enabled equil. to be reached in 24 hrs.; the mixt. then contained 80% II (equil. reached from both sides); when 1% $ZnCl_2$ is used the reaction is slower and even after 900 hrs. II is only 11.3% isomerized, while I is 63.1% isomerized. The results are given graphically. $SnCl_4$ (10%) caused a somewhat less extensive isomerization, but the reaction could not be followed accurately because of tar formation. Use of 10% $AlCl_3$ gave a greater heat evolution on mixing than was observed with the other catalysts, but its isomerization efficiency is poor: after 24 hrs. II is isomerized 11%, and I 43%; 1% $AlCl_3$ is much less effective. If the addn. of butadiene to $ClCH_2OMe$ is conducted using 1% $AlCl_3$ catalyst, the reaction mixt. gives a 30% total yield, composed of 23% II and 77% I. The problem of the possibility of primary addn. being at the 1,2-positions only cannot be solved from the available data; there is an indication that the 1,2-addn. is favored. The metal chloride-catalyzed isomerization probably proceeds similarly to the Friedel-Crafts reaction, involving formation of mesomeric ions of the methoxypentene and $ZnCl_4$-type ions, the isomerization being based on the common resonant ion. The lack of attainment of equil. when low percentages of the catalysts were used is explained by secondary reactions which give products incapable of isomerization. II. **Action of sodium acetate on isomeric methoxychloropentenes and hydrolysis of the resulting acetates.** *Ibid.* 511-8.—The reaction of NaOAc with isomeric methoxychloropentenes is best interpreted on the customary basis of allylic reactions, with the secondary chloride reacting by the S_N^2 reaction mechanism, while the primary chloride reacts by both S_N^2 and to some extent by S_N^1 mechanisms. 1-Methoxy-5-chloro-3-pentene (30 g.), 23 g. AcOH, and 25 g. dry NaOAc heated 2 hrs. at 120-30° gave 2.3 g. *1-methoxy-4-penten-3-ol ace-*

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tate (I), b_9 63-5°, n_D^{20} 1.4264, d_4^{20} 0.9600, and 25.7 g. *1-methoxy-3-penten-5-ol acetate* (II), b_9 82-5°, n_D^{20} 1.4300, d_4^{20} 0.9789. 1-Methoxy-3-chloro-4-pentene (30 g.), 23 g. AcOH, and 25 g. AcONa heated 2 hrs. at 120-30° gave 10.7 g. I and 16 g. II. II (10 g.), 10 g. AcOH, and 10 g. NaOAc heated 2 hrs. at 120-5° produced no change; the same result was obtained with I. I and II were likewise unaffected by boiling with Ac_2O 3 hrs. Hydrolysis of II by hot alc. KOH gave only *1-methoxy-3-penten-5-ol*, b_{10} 97°, n_D^{20} 1.4485, d_4^{20} 0.9523; similar hydrolysis of I gave *1-methoxy-4-penten-3-ol*, b_{10} 60-1°, n_D^{20} 1.4373, d_4^{20} 0.9300. Hydrolysis of the chlorides by alkali also goes without isomerization.

G. M. Kosolapoff

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ARBUZOV, B. A. and Zoroastrova, V. M.

"The Reaction of N,N-dichlorobenzenesulfonamide and Butadiene in Ethanol" Compte Rend Acad Sci URSS, Vol. 5, 1946

"Simultaneous Action of Chlorine and Sodium Alcohols (As well as Chlorine and Acetic Anhydride) on Butadiene" Compte Rend Acad Sci URSS, VOL 53, 1946

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The simultaneous action of chlorine and sodium alcoholate (as well as chlorine and acetic anhydride) on butadiene. B. A. Arbuzov and V. M. Zoroastrova. *Compt. rend. acad. sci. U.R.S.S.* 53, 41-4(1946); cf. *C.A.* 39, 4858*.—A detailed study of the addn. of Et hypohalite and mixed anhydrides of acetic and chloric acids to bivinyl in order to investigate the general phenomena of 1,2- and 1,4-addn. is reported. The following compds. were obtained by repeated fractionation of the products from an alc. soln. of Na satd. with Cl and bivinyl until the resulting soln. was distinctly alk.: 1-chloro-2-ethoxy-3-butene (I), b. 130-9°, n_D^{20} 1.4340, d_4^{20} 0.9784, MR_D 35.80 (theoretical 35.90); 1-chloro-4-ethoxy-2-butene (II), b_{14} 61-4°, n_D^{20} 1.4480, d_4^{20} 1.0070, MR_D 36.76; and (III) a product corresponding by analysis to a compd. formed by the addn. of 2 mols. of EtOCl and 1 of bivinyl, b_8 90-1°, n_D^{20} 1.4530, d_4^{20} 1.1309, MR_D 51.39 (theoretical, 51.27). For identification, I was transformed by alc. alkali into ethyloxyprene, $CH_2{:}C(OEt)CH{:}CH_2$ (IV), b. 94-6°, n_D^{20} 1.4400, d_4^{20} 0.8300, and by the formation of $MeCOCH{:}CH_2$ after the hydrolysis of IV. For identification II was converted by the action of alc. KOH into 1,4-diethoxy-3-butene (V), b_{27} 83°, d_4^{20} 0.9061, n_D^{20} 1.4268, MR_D 41.96 (theoretical 42.15). III is receiving further study to locate the positions of the EtO groups and the Cl atoms. It is certain from the above results, that the entering groups of EtOCl are added to both the 1,2- and 1,4-positions of butadiene. The simultaneous action of Cl and Ac_2O, in the presence of anhyd. NaOAc, on butadiene is likewise reported. Repeated fractions produced the following products: 1-chloro-2-acetoxy-3-butene (VI), b_4 54-6°, b_{760} 164.5-6.5°, n_D^{20} 1.4550, d_4^{20} 1.1300, MR_D 35.68 (theoretical, 35.90); 1-chloro-4-acetoxy-2-butene (VII), b_6 82-4.5°, n_D^{20} 1.4700, d_4^{20} 1.1510, MR_D 36.00; a product (VIII) resulting from the addn. of 2 Cl atoms to VI or VII, b_7 114-16°, n_D^{20} 1.4705, d_4^{20} 1.3049, MR_D 45.63 (theoretical 46.16); and a product (IX), m. 117.8°, resulting from the addition of 2 mols. AcOCl to bivinyl. VI was converted by solid KOH into 1,2-oxybivinyl (X), b. 65-7°, n_D^{20} 1.4220, d_4^{20} 0.8990. VII was not isolated in the pure state but was converted to 2-butene-1,4-diol (XI) and then by NaOAc into the diacetate, $AcOCH_2CH{:}CHCH_2OAc$ (XII), m. 14-16°, b_4 114°, d_4^{20} 1.0842, n_D^{20} 1.4470, MR_D 42.4 (theoretical, 41.98). VIII appears to be derived predominately from VI. The structure of IX is likewise indefinite but appears to result from the simultaneous addn. of a mol. of AcOCl at the 1,2- and 1,4-positions.

The Cl content of II, III, VI, VII, VIII, and IX confirms the former conclusions. R. E. Dunbar

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The reaction of *N,N*-dichlorobenzenesulfonamide and butadiene in ethanol. B. A. Arbuzov and V. M. Zoroastrova. *Compt. rend. acad. Sci. U.R.S.S.* 33, 225-7(1946) (in French).—Contrary to Petrov (*C.A.* 33, 5803[3]), alkyl hypochlorites add to the 1,4- as well as to the 1,2-positions of butadiene (I). The Ingold-Lapworth mechanism for addn. to double bonds is favored with the exception that complete ionization of the entering mole, prior to addn., is not essential. To 250 ml. I in abs. EtOH cooled to −12° was gradually added 159 g. $PhSO_2NCl_2$ (II) so that the temp. was maintained at −6 to −4°. A cryst. ppt. (3 g.) of excess II, m. 58°, was filtered off. After removal of excess I and EtOH by distn., $NaHSO_3$ was added and the reaction product was steam-distd. After ether extn. of the distillate and several refractionations, there were obtained 33 g. *1-chloro-2-ethoxy-3-butene*, b. 133-7°, d_4^{20} 0.9677, n_D^{20} 1.4308; 4 g. *1-chloro-4-ethoxy-2-butene* (III), b_{13} 60-2°, d_4^{20} 1.0037, n_D^{20} 1.4430; 0.9 g. of the addn. product of 2 moles EtOCl to I, b_5 90-1.5°, n_D^{20} 1.4580. Treatment of III with alc. alkali gave *1,4-diethoxy-2-butene*, b_{20} 78.5-9°. Arthur Dolnick

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The parachors and the structure of phosphoric, phosphorus, and phosphinic acid esters. B. A. Arbuzov and V. S. Vinogradova (Univ. Kazan). *Compt. rend. acad. sci. U.R.S.S.* 54, 787-90(1946)(in French).—The parachors of $P(RO)_3$, where R = Ph, Me, Et, Pr, and iso-Bu; $RP:O(OR)_2$, where R = Me, Et, Pr, and Bu; $P(CH_2OCH_2CH_2O)_3$; $(EtO)_3P:S$; and $PhP(OEt)_2$ were measured. Good agreement is found with values calcd. by the method of Gibling (*C.A.* 35, 6493[3]; 39, 3986[9]), correcting for parallelism of the C chains after the β C atoms.

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Bonding agent for sand cores. B. A. Arbusov, M. V. Sobolevskii, I. S. Rez, I. P. Kolobilev, and P. I. Baranov. U.S.S.R. 68,503, June 30, 1947. Cryst. urea is dissolved in formalin contg. urotropin. This mixt. is brought to a boil, cooled, and neutralized with NH_4OH. From the resulting product is sepd., by the usual means, a carbamide resin, and the latter is used as bonding agent.
M. Hosch

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B

Allylic Rearrangement. Part III. The Action of Alcoholic Solutions of Alkali and of Sodium Dithionite on the Isomeric Methoxychloropentenes. (In Russian.) A. N. Pudovik and B. A. Arbuzov. *Bulletin of Academy of Sciences of the U.S.S.R., Section of Chemical Sciences,* July-Aug. 1947, p. 377-386.

Experimental results are given for a number of cases and discussed in terms of reaction mechanisms.

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Parachors and structure of esters of phosphoric, phosphorous, and phosphonic acids. B. A. Arbuzov and V. S. Vinogradova (A. M. Butlerov Research Inst., Kazan State Univ.). *Bull. acad. sci. U.R.S.S., Classe sci. chim.* 1947, 459-72(in Russian); cf. *C.A.* 41, 6096g.—(1) The following esters were synthesized and their *surface tensions*, γ (dyne/cm.), at 20° detd. by the method of max. bubble pressure: $(BuO)_3PO$ 27.79, $(MeO)_3P$ 26.52, $(EtO)_3P$ 24.26, $(PrO)_3P$ 25.91, $(iso\text{-}BuO)_3P$ 23.70, $(PhO)_3P$ 42.46, $(EtO)_3PS$ 29.65, $PhP(OEt)_2$ 31.95, $MePO(OMe)_2$ 36.70, $EtPO(OEt)_2$ 28.94, $PrPO(OPr)_2$ 28.08, $BuPO(OBu)_2$ 27.55, $(MeOCH_2CH_2O)_3P$ 34.79. (2) From γ and the d., the exptl. parachors *P* were detd. and compared with the *P* calcd. by the procedure of Gibling (*C.A.* 35, 6493[1,2]; 37, 1308[1,2]; 39, 235[1], 3986[2]) involving "standard values" (*SV*) for groups and "expansion corrections" (*EC*) depending on the chain length. Using G.'s *SV* = 119.9 for $([C]O)_3PO$ and 149.8 for $([C]O)_3PS$, the calcd. *P* agree with the exptl. *P* for $(PhO)_3PO$ (685.4) and for $(EtO)_3PS$ (431.2) if corrections are applied to allow for the bending of the chains resulting in parallel alignment beyond the β-C atoms and in virtual 5-membered rings O:P.O.C.C. Similar agreement is found for $(iso\text{-}PrO)_3PO$ and $(iso\text{-}BuO)_3PO$. (3) From the exptl. *P* = 267.5 of $(MeO)_3P$, subtraction of 3 × 55.2 (for 3 Me) and of *EC* = 0.66, leaves for $([C]O)_3P$, *SV* = 101.27; with this value, agreement between the calcd. and the exptl. *P* for phosphites is obtained if the same corrections are applied as in the case of phosphates. While in the latter instance the parallel bending of the chains at the β-C atoms could easily be ascribed to polarization of the chains owing to resonance of the P:O bond, in phosphites the same effect can only arise owing to the presence of the electron pair at P; this necessitates assumption of a tetrahedral disposition of valencies around the tricovalent P, with the electron pair occupying the 4th corner of the tetrahedron, and leads to postulation of the existence of optically active antipodes of tricovalent N, P, and As. (4) In the case of phosphates of branched alkyls, the uncertainty as to whether the β-C correction should be applied to only one β-C in each chain or to all, was settled in favor of the latter alternative; e.g., for $(iso\text{-}PrO)_3PO$, the exptl. and calcd. *P* agree only if the correction is made for all six β-C atoms. The available data do not warrant a decision as to whether the correction for parallelism should be applied only to the principal chain or to both principal and lateral chains. For $(MeOCH_2CH_2O)_3P$ it is necessary to correct only for the parallelism of one atom (C or O). (5) From the *P* of phosphonates, applying the same correction for parallelism as in the case of phosphates, one finds for the group $([C]O)_2([C])PO$, *SV* = 93.7. The corrections allowing for the interaction between the resonating P:O bond and the alkyl linked directly to P, are different from those pertaining to the ester alkyls, and, for reasons of analogy, are closer to the corrections applied by Gibling to the bending of chains in esters of carboxylic acids. The structure of phosphonic acid esters resembles closely that of phosphates; it shows 3 virtual 5-membered

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rings O:P.C.C.C. (6) Whereas the discrepancy between the exptl. *P* and the *P* calcd. according to Gibling is mostly 0.2–0.4% and only exceptionally as high as 0.7%, agreement with the *P* calcd. according to Samuel (*C.A.* 38, 3521[1]) is much less satisfactory; in particular, in the case of alkanephosphonic acid esters, the deviation attains 1.5%. Calcn. according to Mumford and Phillips (*C.A.* 22, 1325; 24, 53) gives errors up to 2.5%.

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Allylic rearrangements. IV. Action of the sodium and magnesium derivatives of malonic acid ester and of the sodium derivative of acetoacetic acid ester on isomeric met--chlorpentenes. A. N. Pudovik and B. A. Arbuzov. *Bull. acad. sci. U.R.S.S., Classe sci. chim.* 1947, 501-8(in Russian); cf. *C.A.* 42, 1556g.—Na (4.3 g.) in 120 cc. abs. EtOH treated with 30 g. $CH_2(CO_2Et)_2$, followed by 25 g. $MeOCH_2CH_2CH:CHCH_2Cl$ (I), the mixt. heated on a steam bath 3 hrs., then the EtOH distd., the residue dild. with H_2O and extd. with Et_2O, and the ext. distd., gave 2.5 g. *di-Et 1-methoxy-3-hexene-6,6-dicarboxylate* (II), b_4 132°, n_D^{20} 1.4450, d_4^{20} 1.0213. Na (2.6 g.) in 70 cc. abs. EtOH treated with 18 g. $CH_2(CO_2Et)_2$, followed by 15 g. $MeOCH_2CH_2CHClCH:CH_2$ (III), and worked up as above, yielded 5.8 g. unreacted III, 6.7 g. unreacted $CH_2(CO_2Et)_2$, and 2 g. II. Mg shavings (5 g.), 32 g. $CH_2(CO_2Et)_2$, and 40 cc. abs. EtOH heated to boiling were treated with several drops CCl_4, the formation of the Mg deriv. allowed to proceed with occasional cooling, then the mixt. heated 3 hrs. with stirring until the Mg dissolved, treated dropwise with 28 g. I, and heated 4 hrs. at 110-20°; on cooling, the mixt. was treated with H_2O and HCl and the org. layer sepd. and combined with the Et_2O ext. of the aq. layer; distn. gave 12 g. II ($b_{3.5}$ 130-2.5°) and 14 g. *di-Et 1,11-dimethoxy-3,8-hendecadiene-6,6-dicarboxylate* (IV), $b_{3.5}$ 186-8°, n_D^{20} 1.4590, d_4^{20} 1.0250. A similar reaction using 5 g. Mg, 32 g. $CH_2(CO_2Et)_2$, 40 cc. abs. EtOH, and 28 g. III gave 4.7 g. unreacted III, 10 g. $CH_2(CO_2Et)_2$, 14.7 g. II, and 3.3 g. IV ($b_{3.5}$ 208-10°, n_D^{20} 1.4585, d_4^{20} 1.0259). IV (11 g.) slowly added to 5.5 g. KOH in 50 cc. EtOH and boiled 3 hrs. gave the *free acid*, m. 86-7° (from benzene), purified through the *Ca salt* (by boiling with concd. $CaCl_2$ soln.) and acidification with HCl. Na (5.14 g.) in 50 g. abs. EtOH treated with 29 g. $AcCH_2CO_2Et$, followed by 30 g. I, boiled 3 hrs., freed of EtOH by distn., dild. with H_2O, and extd. with Et_2O, gave on distn. of the ext. 30 g. *Et 1-methoxy-3-octen-7-one-6-carboxylate*, b_{11} 151-3°, n_D^{20} 1.4507, d_4^{20} 1.0047, and 3.5 g. *Et 3-acetyl-1,11-dimethoxy-3,8-hendecadiene-6-carboxylate*, b_4 173°, n_D^{20} 1.4654, d_4^{20} 1.0077. The former ester (9 g.) was added to 2.4 g. KOH in 47 g. H_2O, stirred 2 hrs. at room temp., extd. with Et_2O, and the aq. layer was treated with 8 g. 50% H_2SO_4; warming 10-15 min. on a water bath and extn. with Et_2O gave 5.2 g. *1-methoxy-3-octen-7-one*, b_{11} 102°, n_D^{20} 1.4434, d_4^{20} 0.9149; addn. of the ester (15 g.) to 22.5 g. cooled KOH in 15 g. H_2O and 20 g. EtOH, followed by heating 3 hrs. on a steam bath, gave 1.5 g. of the above ketone (from the Et_2O ext.) and 6.4 g. *1-methoxy-3-hexene-6-carboxylic acid*, b_{11} 158°, n_D^{20} 1.4535, d_4^{20} 1.0174 (from the acidified aq. layer). The latter acid with aq. $KMnO_4$ in the cold (1 hr.) gave methoxypropionic acid, b_{11} 108-9°, n_D^{20} 1.4177. Na (7 g.) in 100 cc. abs. EtOH was treated with 39 g. $AcCH_2CO_2Et$, followed by 39 g. III; after heating 3 hrs. on a steam bath, followed by the usual working up of the org. components, no individual product was isolated and the over-all mixt. obtained b_{10} 35-b_4 200°. Thus, I gave the normally expected reaction products, whereas III apparently reacted in the form of the resonating ion ($MeOCH_2CH_2\overset{+}{C}HCH:CH_2 \rightleftarrows MeO\cdot CH_2CH_2CH:CH\overset{+}{C}H_2$) to give "anomalous" products.

G. M. Kosolapoff

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Action of 1-bromo-1-nitro hydrocarbons on triethyl phosphite. A. E. Arbusov, B. A. Arbuzov, and B. P. Lugovkin. *Bull. acad. sci. U.R.S.S., Classe sci. chim.* 1947, 535-8(in Russian).—$RCHBrNO_2$ react with $(EtO)_3P$ not along the lines of expected formation of $RCH(NO_2)P(O)(OEt)_2$, but anomalously with formation of Et_3PO_4 as the only identifiable product, evidently by a mechanism of the type presented by Allen and Wilson (*C.A.* 34, 3265[1]) for the thermal decompn. of bromonitro derivs. through a biradical mechanism. The over-all reaction may be given by: $CH_3BrNO_2 \rightarrow CH_2\langle + Br + NO_2 + P(OEt)_3 \rightarrow CH_3CH_2 + Et_3PO_4 + NO + Br_2P(OEt)_3$. The aliphatic NO_2 group does not in itself oxidize $(EtO)_3P$. To 11.1 g. $(EtO)_3P$ in 20 cc. Et_2O was slowly added 8.8 g. CH_3BrNO_2 in 20 cc. Et_2O over 20 min.; the temp. rose spontaneously to 40° and, after the Et_2O was distd., the residue was heated to 100°, when a spontaneous reaction raised the temp. to 160° with evolution of 49.2% EtBr; distn. of the residue gave 55% Et_3PO_4 and 3 g. unidentified solid residue; the off-gas produced in the reaction was unsatd. and presumed to be C_2H_4. Similar treatment of Et_3PO_4 with an equimol. amt. of $MeCHBrNO_2$ gave 47% Et_3PO_4. Heating Et_3PO_4 with $MeNO_2$ gave no reaction. To 28.4 g. Et_3PO_4 at 4° was added slowly 22 g. $PhCBr_2NO_2$; spontaneous reaction raised the temp. to 92° and EtBr began to distil; the mass was heated to 180°, when EtBr distn. ceased for a total yield of 74%. Distn. gave 15.5 g. crude Et_3PO_4 and an unidentified product (2.6 g.), b_4 78-81°, n_D^{20} 1.5049, which on standing deposited crystals, m. 237-9° (from EtOAc), free from P and Br and analyzed as $C_{14}H_{12}N_2O$. G. M. K.

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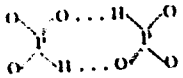
Parachors and the structure of dialkyl phosphorous acids B. A. Arbuzov and V. S. Vinogradova. *Bull. acad. sci U.R.S.S., Classe sci. chim.* 1947, 617-22(in Russian).—In continuation of parachor studies on $(RO)_3P$ and $RPO(OR')_2$ (cf. *C.A.* 42, 3312a), the work was extended to $(RO)_2POH$, with the hope of elucidating the possible tautomeric structures. Using Gibling's method (*C.A.* 39, 238[9]), with the group value of PO_3H being 115.45 (calcd. from *P* of $(MeO)_2POH$), satisfactory agreement was obtained between observed and calcd. values up to the Bu ester, beyond which a const. deviation of 1.1% was observed. Application of a 2.2 unit correction for each C beyond the β-position gives somewhat better agreement, but such doubling of Gibling's standard β-C correction has no theoretical basis, as long as a monomeric structure of the esters is assumed. If a dimeric H-bonded ring structure is assumed, as discussed by Arbuzov, *et. al.* (*C.A.* 41, 3022), the chain interaction correction is applicable to 4 ester chains, i.e., a correction of 8.8 units per every 4 C atoms beyond β-C; i.e., 2.2 units per C atom is explicable. Use of this concept gives the ring-dimer group value for of 220.8 *P* units. The agreement of the calcd. and observed *P* values in this case is excellent up to the octyl ester (highest studied) and the deviations are at most 0.6%, (for iso-Bu ester, if the parallelism correction is made only for the main chains). Use of Samuel's method (*C.A.* 38, 3521[1]) with the calcn. made on the basis of monomeric structures for either tri- or pentavalent P (i.e. $(RO)_3POH$ or $(RO)_2PHO$), led to an av. *P* deviation of 1.1% for P^{IV} forms and of 1.5% for P^{III} forms. Thus, the parachor could not be used to definitely establish the tautomeric configuration of these esters. The dialkyl phosphites were prepd. conventionally from ROH and PCl_3. The following values of surface tension, d., and n were found for the series. $(MeO)_2PHO$, b_{10} 55-5.5°, γ^{20} 37.21, d_4^{20} 1.2004, n_D^{20} 1.4036; $(EtO)_2PHO$, b_{10} 75°, 30.70, 1.0742, 1.4080; $(PrO)_2PHO$, b_{10} 91.5°, 29.21, 1.0184, 1.4008; $(BuO)_2PHO$, b_{10} 115°, 28.53, 0.9888, 1.4230; $(iso\text{-}BuO)_2PHO$, b_{10} 105°, 27.08, 0.9700, 1.4200; $(C_5H_{11}O)_2PHO$, b_5 145-6°, 28.38, 0.9480, 1.4335; $(C_6H_{13}O)_2PHO$, b_5 100-7°, 28.51, 0.9303, 1.4382; $(C_8H_{17}O)_2PHO$, b_5 190-1°, 29.25, 0.9280, 1.4420. It is noteworthy that a min. of surface tension exists between the Bu and Am esters, beyond which surface tension rises with increasing mol. wt. The following dialkyl phosphites are reported as new compds.: $(C_4H_9O)_2PHO$, by addn. of 6.7 g. PCl_3 to 15 g. C_4H_9OH with ice cooling, followed by evacuation to remove HCl and distn. of the product (12 g.) *in vacuo*. $(C_5H_{11}O)_2PHO$, from 8 g. PCl_3 and 13.5 g. ice-cooled $C_5H_{11}OH$, followed by distn. *in vacuo* (yield not stated). $(C_8H_{17}O)_2PHO$, from PCl_3 and ice-cooled $C_8H_{17}OH$; from 18.7 g. PCl_3 there was obtained 18 g. pure ester, b_5 185-7°.

G. M. Kosolapoff



ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, B. A.

USSR/Chemistry - Phosphoric Acid
Chemistry - Ethers, Ethyl

Sep/Oct 1947

"The Action of α -Bromonitrohydrocarbons on the Ethyl Ether of Phosphorous Acid," A. E. Arbuzov, B. A. Arbuzov; B. P. Lugovkin, Chem Sci Res Inst imnei A. M. Butlerov, Kazan State U, 3 pp

"Izv Akad Nauk SSSR, Otd Khim Nauk" No 5

Contains experimental data and results, and shows that in each case where bromonitromethane, α - bromonitroethane and α, α -dibromosphenylnitromethane acts on the ethyl ether of phosphorous acid, the result is triethylphosphate.

PA 53T2

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, B. A.

USSR/Chemistry - Allyl Groups
Chemistry - Esters

Sep/Oct 1947

"Allyl Regroupings," A. N. Fudovik, B. A. Arbuzov, Kazan' State U, 8 pp

"Izv Akad Nauk SSSR, Otd Khim Nauk" No 5

Describes action of sodium and magnesium malonic esters on isomeric methoxychloropentanes, and shows that reaction of esters with a secondary chloride gives same products as with a primary chloride.

PA 53T11

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ARBUZOV, B. A.

IA 53T7

USSR/Chemistry - Parachors
Chemistry - Esters

Sep/Oct 1947

"Parachors and the Structure of Phosphoric Acid, Phosphorous Acid and Phosphinous Acid Esters," B. A. Arbuzov, V. S. Vinogradova, Sci Res Inst imeni A. M. Butlerov, Kazan State U, 13 pp

"Izv Akad Nauk SSSR, Otd Khim Nauk" No 5

Includes data on measurements of parachors of 13 esters of phosphoric, phosphorous and phosphinous acids, and compares them with parachors calculated by Mumford and Phillips and Samuel methods of group values.

53T7

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No. 12

Organophosphorus-tin compounds. I. Synthesis of compounds $R_3SnPO_3R'_3$. B. A. Arbuzov and A. N. Pudovik (Chem. Inst., Acad. Sci., Kazan). *J. Gen. Chem.* (U.S.S.R.) 17, 2158-65(1947).—The Arbuzov reaction was used to prep. compds. of the type $R_3SnPO_3R'_3$ by the interaction of R_3SnX with $P(OR')_3$. The compds. suffer rapid hydrolysis on heating or standing with 5-20% HCl, with rupture of the P—Sn bond; similar cleavage occurs with alk. solns. or with halogens, as well as with AcCl. When 16 g. Me_3SnI and 7 g. $(MeO)_3P$ were heated to 105° a vigorous reaction took place and MeI distd. from the mixt.; the collected distillate was returned and the distn. was repeated at a max. temp. of 110°; 6.2 g. MeI was collected (7.7 g. theoretical); the residue, remaining in the flask, crystd. on standing and represented a 95% yield of *$Me_3SnPO(OMe)_3$*, m. 90° (from EtOH), sol. in Et_2O, $CHCl_3$, Me_2CO, EtOH, and H_2O. This, on heating 3 hrs. with concd. HCl to 100°, gave Me_3SnCl_3, m. 107°; standing at room temp. in 5% HCl gave Me_3SnCl, m. 42°, b_{18} 45-7°; boiling 3 hrs. with distd. H_2O did not change the product, however; treatment with Cl in $CHCl_3$ soln. for 24 hrs. also gave Me_3SnCl, as did similar treatment with AcCl; boiling with 10% aq. KOH gave Me_3SnOH, m. 118°. Heating 23 g. Et_3SnI and 12 g. $(EtO)_3P$ 15 min. to 150° gave 9 g. crude *$Et_3SnPO(OEt)_3$*, which on distn. gave 3 g. pure product, viscous liquid, $b_{1.5}$ 210-20°, n_D^{20} 1.4858; this heated with 20% HCl 10 hrs. at 160-70° in a sealed tube gave Et_3SnCl_3, m. 84.5-85°, while treatment with AcCl and 2 days' standing gave Et_3SnCl, b_{18} 98-100°, and a small amt. of unidentified product, b_5 142-5°, which reduces Fehling soln. $(EtO)_3PONa$, from 4 g. $(EtO)_3POH$, in 30 ml. Et_2O was treated with 8 g. Me_3SnI, boiled 1 hr., the heavy white ppt. sepd., and the Et_2O evapd., to give a yellow oil, which on standing 24 hrs. deposited 0.88 g. *$(Me_3SnOH)_3.Me_3SnI$*, decomp. 145-55°, which apparently was formed from the free radical Me_3Sn, produced in the initial reaction with the sodium phosphite. The $(EtO)_3PONa$ from 4.2 g. $(EtO)_3POH$ in abs. EtOH was treated with 10 g. Bu_3SnI (no NaI pptn. was observed), the EtOH was distd. and was replaced by MeOH, the mixt. boiled, and the soln. decanted from the white solid ppt. and distd. to give a colorless product, b_{11} 83-4°, n_D^{20} 1.4842, d_4^{20} 1.3042, which appeared to be *Et_3SnOEt*. Heating Ph_3SnBr with $(EtO)_3P$ 0.5 hr. to 170° gave only Ph_4Sn, m. 222°; similarly, when $(EtO)_3PONa$ (from 0.26 g. Na) in Et_2O was mixed with 4.5 g. Ph_3SnBr in benzene, heated 2 hrs., freed of Et_2O, and heated 4 hrs. on a steam bath, the filtered soln. on concn. had an odor of $(EtO)_3P$; addn. of Et_2O pptd. 3.7 g. *Ph_4Sn*, m. 222°. Heating Ph_3PbI with $(EtO)_3P$ gave only the disproportionation product, *Ph_4Pb*, m. 224°. Heating 8.5 g. Et_3PbBr and 4.5 g. $(EtO)_3P$ 6 hrs. at 100° gave some $(EtO)_3P$ and a white amorphous solid, insol. in org. solvents. A similar product is obtained on heating a PhMe soln. of Et_3PbBr, apparently a disproportionation product of Et_3PbBr. Thus, the org.-Pb compds. were incapable of the normal Arbuzov reaction. II. **Synthesis of compounds of the type $R_2Sn(PO_3R'_3)_2$.** B. A. Arbuzov and N. P. Grechkin. *Ibid.* 2166-77.—The Arbuzov reaction with R_2SnX_2 gave the expected phosphono-tin compds. of type $R_2Sn(PO_3R'_3)_2$, solids or semisolids, which in most cases could be crystd. from org. solvents. They were sol. in hot BuOH and $CHCl_3$, almost insol. in other solvents, and their crystn. led to severe losses. The high m.p. and poor soly. of the products is explained by their apparent dimeric structure, as shown by Rast mol. wt. detns. of the Et and the Pr compds. The P—Sn bond is readily cleaved on treatment with dil. HCl even at room temp., giving R_2SnCl_2, while HBr gives R_2SnBr_2; 10% NaOH gives R_2SnO, while Cl or Br in $CHCl_3$ soln. give R_2SnX_2. The study was extended briefly to reactions of $RSnX_3$ and SnX_4, which will be reported later. However, the reaction of $MeSnI_3$ with $2(EtO)_3P$ gave, on heating to 85-90°, followed by cooling,

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an abundant crystn. of a golden yellow solid, identified as an intermediate adduct, $MeISn[P(OEt)_3]_3$, which was an addnl. confirmation of the addn. mechanism of the Arbuzov reaction; this adduct m. 101–2° (evolution of EtI). When 20.2 g. Me_3SnI_3 and 12.4 g. $(MeO)_3P$ were heated to 90–100°, a vigorous reaction took place in which 12.5 g. MeI distd. from the mixt.; the glassy residue, dissolved in hot BuOH, gave on cooling 40.6% $Me_3Sn(PO_3Me_3)_3$, m. 245–7°. An analogous procedure was used to prep. the following: $Et_3Sn(PO_3Et_3)_3$, m. 249–51°, 40%; $Pr_3Sn(PO_3Pr_3)_3$, m. 251–3°, 12%; $Et_4Sn(PO_3Pr_3)_3$, m. 202–4°, 10%; $Et_4Sn(PO_3Me_3)_3$, m. 263.5–65°, 43%; $Me_3Sn(PO_3Et_3)$, glassy solid, no m.p. or yield cited. When 4.5 g. $(MeO)_3P$ was slowly added to 6.2 g. $MeSnI_3$ a brisk reaction occurred; the mixt. was heated 10 min. to 60–80°, and the MeI removed *in vacuo* (the wt. loss was equal to theoretical); the residual crumbly white solid was free of halogen, but due to insoly. in org. solvents could not be purified; its behavior with 30% HBr (formation of $MeSnBr_3$ in 50% yield) suggested that it was $MeSn(PO_3Me_3)_3$. Addn. of 4.9 g. $(EtO)_3P$ to 5.5 g. SnI_4, followed by heating to 140° until soln. took place, and removal of EtI *in vacuo* (the wt. loss was equal to theoretical) gave a viscous yellow mass, which could not be purified by crystn. and whose formation of $SnCl_4$ with hot HCl suggested its structure as $Sn(PO_3Et_3)_4$, although satisfactory analyses were impossible because of extremely difficult combustion. An Et_2O soln. of 10 g. $(EtO)_2PONa$ (16 g.), boiled 10 min. with 21.5 g. Et_2SnI_2 in Et_2O, the NaI ppt. filtered off (82.7%), and the filtrate concd., gave 23 g. yellowish liquid which smelled like $(EtO)_3P$; on standing 3–4 hrs. it deposited 7.7 g. of a white amorphous solid identified as Et_2SnO (80%). The washings of the solid (BuOH, EtOH, and Et_2O) on concn. gave a sirup which on long standing deposited 0.8 g. colorless prisms, m. 173–4°, identified as $Et_2Sn(PO_3Et_3)_2$ by analysis and the formation of Et_2SnBr_2 with 30% HBr. This product was a *monomer*, as shown by mol. wt. detn. in $CHCl_3$. Equimol. amts. of $(MeO)_2PONa$ and Me_2SnI_2 gave Me_2SnO as the only identified product (yields Me_2SnCl_2, m. 102–3°, on treatment with HCl). Similarly, $(EtO)_2PONa$ and Et_2SnCl_2 in Et_2O gave 50% Et_2SnO. The oxide is probably the result of atm. O oxidation of the free radical R_2Sn, which is formed in the primary action. Na (1.1 g.) and 6.6 g. $(EtO)_2POH$ in ether with 8.3 g. $MeSnI_3$ immediately formed a ppt. of NaI and when boiled 0.25 hr. gave 97.5% NaI; evapn. of the filtrate gave a mobile liquid yielding, on distn., 1.8 g. mixed $(EtO)_3P$ and $(EtO)_2POH$, after the removal of which the residue decompd. on attempted distn. *in vacuo*; the solid residue was insol. in org. solvents or in HCl (the reaction is still under study at this time).

G. M. Kosolapoff

TRANSLATION: 446940

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ARBUZOV, B. A. and N. P. Grechkin

"Tin-Phosphorus Organic Compounds and Synthesis of Compounds to Type R_2Sn."
Zhurnal Obshchei Khimii, Vol: XVII, No. 12, 1947, pp. 2166-77.

TRANSLATION Available (456199)

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ABUZOV, B. A. (and Pudovik, A. N. (?)

"Obtaining Several Esters of Triaryletheylphosphinic Acid O Poluchanii Nekotorikhovoi Kisioty,"(identified with Pudovik, A. N.) Journ. of Gen. Chem., XVII, 1947, 2193-2148, 2158-2165.

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ARBUZOV, B. A. PA 52T15

USSR/Chemistry - Boric Acid Oct 1947
Chemistry - Esters

"Parachors of Substituted Esters of Boric Acid,"
B. A. Arbuzov, V. S. Vinogradova, Sci Res Inst imeni A. M. Butlerov, Kazan State U imeni V. I. Lenin, 4 pp

"Dok Akad Nauk SSSR" Vol LVIII, No 1

Concludes from data that parachors show the structure of esters of boric acid, which is in complete agreement with data of electro organic research. Favorable concurrence found between calculated and actual significance of parachors.

52T15

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920003-5

ARBUZOV, B. A.

PA 58T11

USSR/Chemistry - Phosphoric Acid, Dialkyl Jan 1947
Chemistry - Parachors

"Parachor and Structure of Dialkylphosphoric Acid," B. A. Arbuzov, V. S. Vinogradova, Sci Res Chem Inst imeni A. M. Butlerov, Kazan State U, 2 pp

"Dok Akad Nauk SSSR, Nova Ser" Vol LV, No 1

Describes experiments which produce data on parachors of dialkylphosphoric acid, fully confirming results on polymerizing ability of this acid in liquid condition.

58T11

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