"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

Mbr. , Scientific Research Institute of Chemistry im. Butlerov, Kazan State University, -1946-

"On the Complements and the Structure of the Ethers of Boric Acid," Dok. AN, 55, No. 5, 1947

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1ST AND 2ND ORDERS

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3RD AND 4TH ORDERS

The parachors and structure of the alkoxyphosphorous acids. B. A. Arbusov and V. S. Vinogradova (Univ. Kazan). Compt. rend. acad. sci. U.R.S.S. 55, 31-2 (1947)(in French); cf. C.A. 41, 6096g.—The following parachors were observed for the designated alkoxyphosphorous acids: dimethoxy 226.3; diethoxy 302.0; di-n-isopropoxy 378.9; diisopropoxy 377.8; dibutoxy 454.7; diisobutoxy 453.1; dihexoxy 607.5; diheptoxy 686.0; dioctoxy 764.0. Parachors calcd. correcting only for deformation by C in β position and for parallelism of chains beyond the β C gave errors of 0 to −1.1%. Consideration of the dimerism of these acids gave calcd. values in error by 0 to −0.6%. Cyrus Feldman

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ARBUZOV, B. A.

USSR/Chemistry - Phosphoric Acids, Esters Jul 1947
Chemistry - Synthesis

"Synthesis of the Esters of Tin Trialkyl-Phosphone," B. A. Arbuzov, Corr Mem, Acad Sci; A. N. Pudovik, Chem Inst Kazan Br, Acad Sci USSR, 2 pp

"Dok Akad Nauk SSSR, Nova Ser" Vol LVII, No 2

Reaction for formation of esters of alkylphosphonic acids in metallo-organic unions, containing haloids. Presents some basic facts concerning chemical reactions. Describes tests which produced subject esters as a result of tin trialkyl-haloid and trialkylphosphite relationship. Submitted, 14 May 1947.

60T1

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ARBUZOV, B. A. 53T18

USSR/Chemistry - Phosphoric Acid Aug 1947
Chemistry - Synthesis

"Synthesis of Diphosphondialkyl Esters," B. A. Arbuzov, Corr Mem Acad Sci USSR; N. P. Grechkin, Chem Inst, Kazan Br, Acad Sci USSR, 3 pp

"Dok Akad Nauk SSSR, Nova Ser" Vol LVII, No 4

Gives data collected during studies on synthesis of diphosphondialkyl esters, and, in particular, triphosphonalkyl esters usually found in compounds such as $R_2Sn\ Hal_2$ or $RSnHal_3$ with complete phosphoric acid esters. Submitted, 14 May 1947.

[redacted] 53T18

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CA

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Transition of alloöcimene on activated carbon. B. A. Arbuzov and A. B. Vlichinskaya. *Doklady Akad. Nauk S.S.S.R.* 57, 785-8(1947); *Chem. Zentr.* (*Russian Zone Ed.*) 1948, II, 1420; cf. *C.A.* 39, 1407[9].—In the course of an investigation of the transitions undergone by α-pinene on activated C at 360-80°, it was necessary to study the behavior of alloöcimene (I) under the same conditions since α-pinene is converted into I at 360-80°. I b_{10} 72°, d_4^{20} 0.8115, n_D^{20} 1.5435. The tube used was 55 cm. long and 2 cm. in diam., the C layer 45 cm. deep, and the rate of flow 5-8 g./hr. The I was preheated to 320°. Marked carbonization was observed and liquid products were formed which amounted to about 55-60% of the I. The C was soon exhausted and had to be renewed. Fractionation of the reaction products yielded m-xylene, along with *2,6-dimethyloctane* (II), b_{760} 159-60°, d_4^{20} 0.7405, n_D^{20} 1.4100, mol. refraction 48.48, and *1,2,3-$C_6H_3Me_3$* (III). Since I is converted into α- and *β-pyronene* by heating to 380-400°, it is assumed that when I is heated with activated C, a portion is reduced to II and, in addn., III and finally m-xylene are formed by the splitting off of the Me group from pyronene. The heating of the mixt. of α- and β-pyronene with activated C (as when I is so heated) yielded m-xylene, III, and a hydrocarbon having properties similar to those of 1,2-dimethyl-3-isopropylcyclopentane; it b_{760} 159-60.5°, d_4^{21} 0.7877, n_D^{21} 1.4328, mol. refraction 46.23. M. G. Moore

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ARBUZOV, B.A., redaktor; DOLGOPOLOSK, B.A., redaktor; KARGIN, V.A., redaktor; MEDVEDEV, S.S., otvetstvennyy redaktor; RAFIKOV, S.R., redaktor; ROGOVIN, Z.A., redaktor; VASKEVICH, D.N., redaktor izdatel'stva; SIMKINA, Ye.N., tekhnicheskiy redaktor

[Proceedings of the third conference on high molecular weight compounds; polymerization and polycondensation] Trudy tret'ei konferentsii po vysokomolekulyarnym soedineniiam; polimerizatsiia i polikondensatsiia. Moskva, Izd-vo Akademii nauk SSSR, 1948. 177 p. (MLRA 10:1)

1. Konferentsiya po vysokomolekulyarnym soyedineniyam. 3d, Moscow, 1945.
(Polymerization) (Condensation products (Chemistry))

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Allylic rearrangements. V. Action of phenylmagnesium bromide on isomeric methoxychloropentenes. A. N. Pudovik and B. A. Arbuzov. *Bull. acad. sci. U.R.S.S., Classe sci. chim.* 1948, 246–9; cf. *C.A.* 42, 1887*b*.—To the PhMgBr from 6 g. Mg and 35 g. PhBr in ether was added with cooling 30 g. $MeOCH_2CH_2CH{:}CHCH_2Cl$; after heating 1 hr., the mass was treated with ice and HCl to give 7.7 g. *1-methoxy-3-phenyl-4-pentene* (I), b_4 100–8°, n_D^{20} 1.5032, d_4^{20} 0.9300, and 17 g. *1-methoxy-5-phenyl-3-pentene* (II), $b_{4.5}$ 103–4°, n_D^{20} 1.5125, d_4^{20} 0.9467. A similar reaction with $MeOCH_2CH_2CHClCH{:}CH_2$ gave 8.8 g. I and 15.5 g. II. Oxidation of I by $KMnO_4$ gave BzOH and *α-phenyl-γ-methoxybutyric acid*, b_{11} 175–8°. Oxidation of II gave BzOH, $PhCH_2CO_2H$, and *β-methoxypropionic acid*, b_{11} 108–12°, n_D^{20} 1.4190. The results indicate that the reaction proceeds by ionic mechanism through the intermediate ion, $[MeOCH_2CH_2\overset{+}{C}H{:}CHCH_2 \leftrightarrow MeOCH_2CH_2\overset{+}{C}HCH{:}CH_2]Cl^-$. G. M. Kosolapoff

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1ST AND 2ND ORDERS

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26

Parachors and Structures of Boric Acid Esters. (In Russian.) B. A. Arbuzov and V. S. Vinogradova. *Zhurnal Fizicheskoi Khimii* (Journal of Physical Chemistry), v. 21, Mar. 1948, p. 303-309.

Describes synthesis of three of the above esters not previously reported. Gives data on molecular parachors of six of these esters, and discusses structural considerations. 12 ref.

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ARBUZOV, B. A.

PA 65T21

USSR/Chemistry - Boric Acid, Esters of Mar 1948
Chemistry - Parachors

"Parachors and Structure of the Esters of Boric Acid," B. A. Arbuzov, V. S. Vinogradova, Chem Sci Res Inst, Kazan State U, 6 pp

"Zhur Fiz Khim" Vol XXII, No 3

Measure parachors of six esters of boric acid. Compare these values with values calculated by the Gibling "group values" method. Variation between the two does not exceed 0.5%. Make conclusions on the structure of boric acid esters on the basis of the parachor findings. Submitted 16 Apr 1947.

[redacted]

65T21

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

USSR/Chemistry - Acids, Manufacture of Organic
Chemistry - Synthesis

1 Mar 1948

"Synthesis of Substituted Benzylphosphine Acids and Their Esters," B. P. Lugovkin, B. A. Arbuzov, Corr Mem Acad Sci USSR, Sci Res Chem Inst imeni A. M. Butlerov, Kazan State U imeni V. I. Ul'yanov-Lenin 3½ pp

"Dok Akad Nauk SSSR, Nova Ser" Vol LIX, No 7

Since very diverse benzyl derivatives substituted in the benzene nucleus easily obtained by the reaction of chlorine methylation, possible with the reaction with full esters of phosphorous acid or dialkylphosphorous sodium to synthesize esters of corresponding substituted benzylphosphine acids. Thus with the action of triethylphosphite on substituted derivatives of benzyl chloride, authors synthesized esters of benzylphosphine acid which contains alkyl groups, haloids, oxygen group, nitrogen group, and aldehyde group in the phenyl radical.

PA47T12

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

PA47T17

ARBUZOV, B. A.

USSR/Chemistry - Phosphorous Compounds, Organic Mar 1948

Chemistry - Silicon

"Silicon Phosphororganic Derivatives," B. A. Arbuzov, A. N. Pudovik, Chem Inst, Kazan Branch, Acad Sci USSR, 3 pp

"Dok Akad Nauk SSSR, Nova Ser" Vol LIX, No 8

Describes experiment designed to examine the reaction between triethylphosphite and haloid silicon phosphor-organic derivatives: led to obtaining compounds with phosphorous-silicon connection.

47T17

[redacted]

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Synthesis of substituted α-toluenephosphonic acids and their esters. B. P. Lugovkin and B. A. Arbuzov. *Doklady Akad. Nauk S.S.S.R.* 59, 1301-4(1948).—Heating $ClCH_2$ derivs. with $(EtO)_3P$ 1-3 hrs. at 150-200° until EtCl evolution stopped resulted in the formation of a no. of corresponding di-Et phosphonates and tetra-Et diphosphonates; the former were insol. in water, the latter were sol. The following compds. were prepd. (yields in parentheses): *4-$MeC_6H_4CH_2PO(OEt)_2$*, b_2 160-3°, n_D^{20} 1.4968, d_{20}^{20} 1.0832 (81%) (*free acid*, m. 185-6°); *1,4-$C_6H_4(CH_2PO_3Et)_2$*, b_2 204°, m. 72-3° (43%) (*free acid*, m. 208-71°); *2,5-$Me(CHMe_2)C_6H_3CH_2PO(OEt)_2$*, b_2 146-8°, n_D^{20} 1.4940, d_{20}^{20} 1.0420 (61%) (*free acid*, m. 175-7°); *3,4-$Me_2C_6H_3CH_2PO(OEt)_2$*, b_2 130-2°, n_D^{20} 1.4990, d_{20}^{20} 1.0708 (91%) (*free acid*, m. 184-6°); *3,6,1,3-$Me_2C_6H_2(CH_2PO_3Et_2)_2$*, b_2 192-4°, n_D^{20} 1.4965, d_{20}^{20} 1.1383 (74%) (*free acid*, m. about 264°); *3,6,1,4-$Me_2C_6H_2(CH_2PO_3Et_2)_2$*, m. 58-9° (87%) (*free acid*, m. 340-60°); *di-Et 3,4-tetramethylene-α-toluenephosphonate* (or *di-Et tetrahydronaphthylmethanephosphonate*), b_2 165-7°, n_D^{20} 1.5165, d_{20}^{20} 1.1063 (68%) (*free acid*, m. 165-6°); *di-Et 6-chloro-8-phosphonomethyl-1,3-benzodioxane*, b_2 195-8°, n_D^{20} 1.5230, d_{20}^{20} 1.2892 (89%); *2,5-$HO(O_2N)C_6H_3CH_2PO(OEt)_2$*, m. 137° (76%) (*free acid*, m. 224-9°); *2,5-$MeO(OHC)C_6H_3CH_2PO(OEt)_2$*, b_2 184-6°, n_D^{20} 1.5280, d_{20}^{20} 1.1921 (70%) (*phenylhydrazone*, m. 117°). The free acids were obtained in 85-90% yields by hydrolysis of the esters with dil. HCl in sealed tubes 5 hrs. at 130-50°. Attempted prepn. of p-$O_2NC_6H_4CH_2PO(OEt)_2$ from p-$O_2NC_6H_4CH_2Cl$ failed and $(EtO)_3P$ was converted to Et_3PO_4. Cf. *C.A.* 40, 1443[1]. G. M. Kosolapoff

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ARBUZOV, B. A.

Action of halo-substituted derivatives of ethylene on butadiene and anthracene. V. M. Zoroastrova and B. A. Arbuzov. *Doklady Akad. Nauk S.S.S.R.* 60, 59–61 (1948).—Anthracene reacts with haloethylenes to give diene adducts, which are apparently bicyclic in nature, with the bridge across the 9,10-positions of anthracene. Anthracene (5 g.) and 20 ml. $(ClCH:)_2$ heated in a sealed tube 10 hrs. at 150–60° and 8 hrs. at 200–20°, filtered from 1.25 g. unreacted anthracene, and concd. yielded 4.5 g. of an *adduct*, $C_{18}H_{12}Cl_2$, m. 203–4° (from ClCH:CHCl). Anthracene (4 g.) and 15 ml. $ClCH:CCl_2$ after 24 hrs. at 200–20° similarly gave 2.2 g. *adduct*, m. 140° (from $CHCl:CCl_2$). Anthracene (5 g.) and 15 ml. $(Cl_2C:)_2$ heated 62 hrs. at 200–30° gave much HCl, which indicated loss of HCl from the original adduct, and an *adduct*, $C_{16}H_8Cl_2$, m. 179–80° [from $(ClCH_2)_2$], which has active double bonds, as shown by the action of *maleic anhydride*, which gave a product m. 255° (from benzene-petr. ether). Heating 2 g. anthracene and an equiv. amt. of $AcOCH_2CH:CHCH_2OAc$ 28 hrs. at 200–20° gave 1.1 g. of an *adduct*, $C_{22}H_{22}O_4$, m. 108–9° (from EtOH). Butadiene (15 g.) and 40 g. $(ClCH:)_2$ heated 5 hrs. at 160–75° and 7 hrs. at 200–10° in the presence of hydroquinone gave 1.5 g. adduct, $C_6H_8Cl_2$, m. 43° (from EtOH), b. about 135–6°, which is probably *1,2-dichloro-4-cyclohexene*.

G. M. Kosolapoff

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CHEMISTRY AND PROPERTIES MOLS

Parachors and structure of esters of orthoformic, orthocarbonic, and orthosilicic acids. II. A. Arbuzov and V. S. Vinogradova (Kazan State Univ., U.S.S.R.). *Doklady Akad. Nauk S.S.S.R.* 60, 799-802(1948).—The parachors P of the following esters were measured: esters of $HC(OH)_3$: Me, 249.0, Et, 367.1, Pr, 483.3, iso-Pr, 481.5, Bu, 599.2, iso-Bu, 598.7, iso-Am, 714.6. Esters of $C(OH)_4$: Me, 290.3, Et, 456.2, Pr, 612.6, Bu, 769.1; attempts to synthesize esters with higher alkyls, by the action of alcoholates on chloropicrin, failed. Esters of $Si(OH)_4$: Me, 328.0, Et, 485.3, Pr, 639.9, Bu, 799.3, $(n\text{-}C_6H_{13})_4$ 1114.1, $(n\text{-}C_7H_{15})_4$ 1278.8, $(n\text{-}C_8H_{17})_4$ 1441.8, $(n\text{-}C_9H_{19})_4$ 1591.8, $(C_{10}H_{21})_4$ 1755.4. For the orthoformates, exptl. data are in good agreement with those calcd. (P = 83.82 is used for the group $HC[O(C)]_3$, and corrections are applied for the bend at the γ-C atom and for parallelism of all 3 chains beyond the γ-C atom); the deviations are at the max. 0.5-0.6%. In the case of the orthocarbonates, there is agreement with the calcd. values if P for the group $C[O(C)]_4$ is taken = 77.64 and if the same corrections are applied as previously. However, one finds the same agreement (within 0.1%) between the exptl. and the calcd. P for $C(OBu)_4$ whether one corrects for parallelism of 3 chains or for 2 pairs of parallel chains. Data for orthocarbonates do not warrant a decision between the 2 alternatives. On the other hand, the exptl. values for the orthosilicates are in distinctly better agreement with calcns. (with P = 106.2 for $Si[O(C)]_4$) based on the assumption of 2 pairs of parallel chains, rather than 3 parallel chains. By analogy, the same structure is also probable for the orthocarbonates.

N. Thon

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Chief-Korrespondent
of Acad. of Sciences USSR (for Alkyl)
Kharkov Chem. Inst.
A. M. Butlerov

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

Parachor and structure of amines. U. A. Arbuzov and L. M. Guzhavina (Kazan State Univ.). *Doklady Akad. Nauk S.S.S.R.* 61, 63–6(1948).—(1) Newly detd. values of the parachor P are: $BuNH_2$ 218.0, n-$C_6H_{11}NH_2$ 370.8, Et_2NH 219.2, Pr_2NH 298.0, Bu_2NH 377.3, $(n$-$C_6H_{11})_2NH$ 845.7, Pr_3N 418.8, Bu_3N 531.3, $(n$-$C_6H_{11})_3N$ 1007.2, $(n$-$C_7H_{15})_3N$ 1240.0, $(n$-$C_{11}H_{23})_3N$ (2.27 mole % soln. in C_6H_6) 1987.6, $(MeOCH_2CH_2)_3N$ 475.5, $(PrOCH_2CH_2)_3N$ 707.9. These data, and the previously known P of $MeNH_2$, $EtNH_2$, $PrNH_2$, Me_2NH, and Et_3N, are in very good agreement with values calcd. on the assumption of the group parachors $(C)NH_2$ 44.5, $(C)_2NH$ 29.7, and $(C)_3N$ 13.0; deviations are mostly 0.0–0.3%, and rarely attain 0.0–0.7%, if corrections are applied for bends of the chains at the γ-C atom and for chain parallelism beyond the γ-C atom. In the case of the tertiary amines, calcns. of P made on the assumption of all 3 chains parallel and pointing in the same direction, and of 2 chains in one direction and the 3rd chain in the opposite direction, show that only the latter structure is consistent with the data; this is in agreement with the conclusion of Staudinger and Rössler (*C.A.* 30, 2460[8]) from viscosity data. No such definite conclusion can be drawn with regard to secondary amines, but comparison of the exptl. P for $(n$-$C_6H_{11})_2NH$ with the P calcd. on the assumption of the 2 chains pointing in opposite direction, and of both chains parallel, speaks rather in favor of the 2nd alternative, and against the conclusion of S. and R. from viscosity. That the configurations in the pure liquid state, and in soln. in C_6H_6, are not different, is demonstrated by the independence of P of the state for $(n$-$C_6H_{11})_3N$ and $(n$-$C_7H_{15})_3N$. (2) The consts. of the newly synthesized *tris(propoxyethyl)-amine*, $(PrOCH_2CH_2)_3N$, are b_{17} 150°, n_D^{20} 1.4377, d_4^{20} 0.9014, γ_{20} 28.82.

N. Thon

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ARBUZOV, B. A. , T. G. Shavsha, V.S. Vinogradova and L. M. Guzhavina

"Application of parachores and dipole moments to the study of the intimate structure of certain organic compounds of phosphorus, boron, nitrogen, and silicon," for which the authors were awarded the 1948-49 D. I. Mendeleyev Prize and 20,000 rubles.

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Allylic rearrangements. IX. Action of sodium salts of dialkyl phosphorous acids and esters of phosphorous acid on isomeric methoxychloropentenes. A. N. Pudovik and B. A. Arbuzov (Lab. Org. Khim., Kazan Gosudarst. Univ.). *Izvest. Akad. Nauk S.S.S.R., Otdel Khim. Nauk* 1949, 522-38; cf. *C.A.* 43, 5737a.—The reactions of $(RO)_2PONa$ with $MeOCH_2CH_2CH:CHCH_2Cl$ (I) proceed normally without allylic rearrangement. Addn. of 39 g. I to $(EtO)_2PONa$ [from 40 g. $(EtO)_2POH$ and 6.6 g. Na in 300 ml. Et_2O] and heating 3 hrs. gave, upon filtration, 5 g. I, 8 g. unknown matter, b_{10} 90-127°, and 38 g. *1-methoxy-5-diethylphosphono-3-pentene* (II), b_{10} 146-7°, n_D^{20} 1.4480, d_4^{20} 1.0380, sol. in H_2O, and extractable by Et_2O unchanged. Heating the ester (10 g.) 3 hrs. to 120° with concd. HCl in a sealed tube, followed by release of the RCl, reheating 6 hrs., and repeated evapn. of the soln. with H_2O gave an oil, $C_5H_{13}O_4P$, apparently $MeOCH_2CH_2CH:CHCH_2PO_3H_2$, which gave a poorly sol. *Ca salt*; II added almost the theoretical amt. of Br in $CHCl_3$; oxidation of II with $KMnO_4$ (22 g. per 12 g. ester) at 10-12° gave 3.6 g. $MeOCH_2CH_2CO_2H$, b_{10} 106-8°, n_D^{20} 1.4184; similarly oxidation of II with peroxyphthalic acid gave nearly theoretical results. A similar reaction of 20 g. I with $(iso\text{-}BuO)_2PONa$ (from 3.5 g. Na and 30 g. ester) in 200 ml. Et_2O gave 5 g. I and 20 g. *1-methoxy-5-diisobutylphosphono-3-pentene* (III), b_1 145-6°, n_D^{20} 1.4455, d_4^{20} 0.9819. The reaction with $(MeO)_2PONa$ was slow because of poor soly. of the Na deriv. in Na; prolonged warming of 20 g. $(MeO)_2POH$ with 4.2 g. Na in 200 ml. Et_2O with stirring (some Na was unreacted), followed by addn. of 24 g. I and heating 10 hrs., gave little action and almost all the I was recovered (1.2 g. product, b_{10} 136-40°, was isolated); addn. of 20 g. $(MeO)_2POH$ to 4.3 g. Na in 50 ml. MeOH, followed by 25 g. I, after cooling, and heating 3 hrs. on a steam bath gave 14.4 g. *1,5-dimethoxy-3-pentene*, b_{10} 57-8°, n_D^{20} 1.4272, d_4^{20} 0.8840, and 14.3 g. *1-methoxy-5-dimethylphosphono-3-pentene*, b_{10} 144°, n_D^{20} 1.4530, d_4^{20} 1.0950. Reaction of I with $P(OR)_3$ also proceeds normally. Thus, 12 g. $(MeO)_3P$ and 13 g. I after 5 hrs. at 140-50° in a sealed tube gave 9.1 g. of the above ester, b_{10} 140-1°, and much unreacted I and $(MeO)_3P$; similarly, 14 g. $(EtO)_3P$ and 11.3 g. I after 10 hrs. at 140-50° gave 7.1 g. unreacted [illegible] II, b_4 133-4°, n_D^{20} 1.4480, d_4^{20} 1.0385. Similar reaction of $(RO)_3P$ with $MeOCH_2CH_2CHClCH:CH_2$ (IV), however, yield the same products as those obtained with I; the reactions are slow and incomplete and the high temps. required do not permit detg. of whether or not there occurs an allylic rearrangement or the usual isomerization on heating which takes place with I and IV. Thus 14 g. $(EtO)_3P$ and 11.3 g. IV, after 10 hrs. at 140-50°, gave 20 g. unreacted materials and but 3.1 g. II, b_{10} 148-9°, n_D^{20} 1.4480, while 12 g. $(MeO)_3P$ and 13 g. IV, after 5 hrs. at 140-50°, gave but 1.4 g. of the *dimethylphosphono analog*, b_4 140-2°, n_D^{20} 1.4530. The course of the reactions of IV with $(RO)_2PONa$ depends upon the presence or absence of excess free $(RO)_2POH$; in the 1st instance the reaction proceeds with complete allylic shift and yields 60-80% $MeOCH_2CH_2CH:CHCH_2PO(OR)_2$ (described above) and no isomers could be detected; in the 2nd instance, however, the reactions yielded as the main product high-boiling substances which were shown to be diphosphonates of the type $MeOCH_2CH_2CH_2CH(PO_3R_2)CH_2CH_2CH_2PO_3R_2$ and $MeOCH_2CH_2CH_2CH(PO_3R_2)CH_3$, PO_3R_2, apparently formed by the 2 possible modes of addn. of $(RO)_2PONa$ to the double bond of the primary compds. of type II or III, and the adducts, reacting with unused IV, yield the above diphosphonates, NaCl, and methoxypentadiene (which was isolated in all cases). Thus, addn. of 40 ml. dry C_6H_6 and 14 g. IV to $(EtO)_2PONa$ (from 2.5 g. Na and 30 g. $(EtO)_2POH$) in Et_2O, followed by distn. of the Et_2O (the reaction is too slow at the b.p. of Et_2O) and refluxing 3 hrs., gave upon filtration of the NaCl, 3.1 g. IV, 12.8 g. $(EtO)_2POH$, 1.9 g. intermediate fraction, and 12.6 g. II, b_{10} 144-7°; repetition with 40 g. $(EtO)_2POH$, 3.3 g. Na, and 19 g. IV gave similarly 14.8 g. II, b_{10} 150-1°. Similarly, 30 g. $(iso\text{-}BuO)_2POH$, 1.7 g. Na, and 10 g. IV in C_6H_6 gave 2.8 g. IV, 14.3 g. $(iso\text{-}BuO)_2POH$, 1.7 g. intermediate fraction, and 11.5 g. III, b_{10} 173-5°. A similar reaction of 30 g. $(EtO)_2POH$, 5 g. Na, and 25 g. IV in C_6H_6 (3 hrs. refluxing), gave 11 g. NaCl, 4 g. IV, 3 g. $(EtO)_2POH$, 13.8 g. (probably) *MeOCH₂CH₂CH₂CH(PO₃Et₂)CH₂PO₃Et₂* (IX), b_{10} 222°, b_1 210-17°, n_D^{20} 1.4478, d_4^{20} 1.1000, and 14 g. glassy residue. Repetition with 8.5 g.

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K instead of Na (6 hrs. refluxing) gave 3.6 g. $MeOCH_2CH:CHCH:CH_2$, 1.6 g. IV, 14.3 g. of the above diphosphonate, b_{10} 214-16°, n_D^{20} 1.4490, 1.6 g. diphosphonate, probably $MeOCH_2CH_2CH(PO_3Et_2)CH_2CH_2PO_3Et_2$ (X), b_{10} 238-46°, n_D^{20} 1.4600, and 7.5 g. undistillable residue. IX is a viscous greenish liquid, sol. in org. solvents and H_2O, unchanged after 1 hr. in H_2O at 100° or after 3 hrs. at 150-60° with MeI, forms no adducts with CuCl, does not add Br, and is not oxidized by peroxyphthalic acid; heating with concd. HCl 2 hrs. at 120°, and reheating 6 hrs. after release of the HCl, gave on evapn. with H_2O a sirup which was a tetrabasic acid (7 g. required 5.8 g. KOH for neutralization). A similar reaction of 29 g. $(iso\text{-}BuO)_3POH$, 3.5 g. Na, and 20 g. IV gave 4.8 g. methoxypentadiene, 8.3 g. liquid, b_7 100-20°, and 15.3 g. *tetra-iso-Bu analog* of IX, b_7 225-9° (crude), b_{10} 237°, n_D^{20} 1.4470, d_4^{20} 1.0203. $(MeO)_3POH$ 20 g., 4.3 g. Na, and 25 g. IV, even after 7 hrs. refluxing, gave largely unreacted materials and only 0.4 g. methoxypentadiene and 3.2 g. substance, b_{10} 50-80°. No reaction took place on heating 10 g. II with 12 g. $(EtO)_3POH$ in the presence of a small amt. of either MeONa or $(EtO)_3PONa$ 3 hrs. to 150° in a sealed tube. To $(EtO)_3PONa$ (from 12 g. $(EtO)_3POH$ and 2 g. Na in Et_2O) was added 40 ml. C_6H_6 and 20 g. II, Et_2O distd., and the mixt. treated with stirring and heating with 12 g. IV and refluxed 3 hrs. (pptn. of colloidal NaCl); distn. gave 1.5 g. methoxypentadiene and 2.7 g. IV, and the viscous residue, treated with H_2O and Et_2O and the org. layer distd., gave 2.1 g. liquid, b_{11} 140-215°, 8.3 g. IX, b_{11} 215-18°, n_D^{20} 1.4495, and 4.9 g. X, b_{10} 235-45°, n_D^{20} 1.4505.—When $(EtO)_3PONa$ (from 12 g. $(EtO)_3POH$ and 2 g. Na in Et_2O) was similarly heated in CaH_2 3 hrs. with 20 g. II and 12 g. I, there was obtained only 28 g. II and no other products. A similar 3-hr. reaction of $(iso\text{-}BuO)_3PONa$ (from 15 g. $(iso\text{-}BuO)_3POH$ and 1.8 g. Na) with 22 g. III and 10 g. IV gave 2.4 g. methoxypentadiene, 1.7 g. IV, 5.1 g. intermediate fraction, b_7 110-220°, and 11.6 g. *tetra-iso-Bu analog* of IX, b_7 236-8°, n_D^{20} 1.4470. Similarly $(EtO)_3PONa$ (from 6 g. $(EtO)_3POH$ and 1 g. Na) and 10 g. II in CaH_2 on distn. of the Et_2O and part of CaH_2 gave 1.1 g. product, b_{10} 214-16°, n_D^{20} 1.4478 (apparently IX), 1.3 g. intermediate fraction, and 3.1 g. X, b_{10} 237-43°, n_D^{20} 1.4500.

X. Action of alcoholic solutions of alkali on isomeric butoxychloropentenes. A. N. Pudovik and V. I. Nikitina. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 19, 75-82(1949).—The reaction of KOH with *1-butoxy-5-chloro-3-pentene* (I) in MeOH or RO derivs.; in PrOH, BuOH, and *tert*-BuOH there is concurrent formation of *1-butoxy-2,4-pentadiene* (III) and dibutoxypentenyl ether by the monomol. S_N1 route, made possible by lesser dissocn. of these alcs. into RO and a proton. With *1-butoxy-3-chloro-4-pentene* (II), both S_N1 and S_N2 mechanisms are operative. Boiling 15 g. I and 7 g. KOH in 80 ml. MeOH 3 hrs. gave 9 g. *1-butoxy-5-methoxy-3-pentene*, b_{10} 93°, n_D^{20} 1.4328, d_4^{20} 0.8086; 20 g. I and 10 g. KOH in 100 g. EtOH gave 12.7 g. *1-butoxy-5-ethoxy-3-pentene*, b_{10} 96-7°, n_D^{20} 1.4336, d_4^{20} 0.8677; 18 g. I and 9 g. KOH in 80 g. PrOH gave 0.6 g. *1-butoxy-2,4-pentadiene*, b_{10} 59-60°, n_D^{20} 1.4490, d_4^{20} 0.8230, and 10.8 g. *1-butoxy-5-propoxy-3-pentene*, b_{10} 110°, n_D^{20} 1.4342, d_4^{20} 0.8606; 30 g. I and 20 g. KOH in 200 g. BuOH gave 4.2 g. III, b_{10} 65°, 10.3 g. *1,5-dibutoxy-3-pentene* (IV), b_{10} 116°, n_D^{20} 1.4352, and 1.3 g. *bis(butoxypentenyl) ether*, b_7 180°, n_D^{20} 1.4535; 20 g. I and 10 g. KOH in 120 g. *tert*-BuOH gave 4.7 g. III and 5.9 g. bis(butoxypentenyl) ether, as well as 5.2 g. intermediate unseparable mixt.; 27 g. I and 14 g. KOH in 200 g. dioxane after 9 hrs. gave 6 g. III and 1 g. of the above ether, as well as 12.5 g. I. Heating 20 g. I with 10 g. KOH in 100 g. 70% EtOH 3 hrs. gave 11.8 g. *1-butoxy-5-ethoxy-3-pentene* (V), b_{10} 107-9°, and 2 g. *1-butoxy-3-penten-5-ol*, b_{10} 125-6°, n_D^{20} 1.4480, d_4^{20} 0.9039. I (40 g.) and 5.5 g. Na in 195 g. BuOH after 4 hrs. at 100° gave 14.5 g. III and 6.2 g. IV, b_{10} 121°, n_D^{20} 1.4393, d_4^{20} 0.8583. Heating 9 g. I and 5 g. powd. KOH 7 hrs. on a steam bath gave 0.8 g. III and 5.5 g. I. II (40 g.) and 20 g. KOH in 120 g. EtOH refluxed 3 hrs. gave 10.2 g. III, 9.3 g. *1-butoxy-3-ethoxy-4-pentene*, b_{10} 78°, n_D^{20} 1.4280, d_4^{20} 0.8032, and 3.2 g. V. Heating 2 g. maleic anhydride and 3 g. III gave a liquid adduct, $C_{13}H_{18}O_4$, b_{11} 192°.

XI. Action of isomeric methoxychloropentenes on sodioalkylacetoacetic esters. A. N. Pudovik. *Ibid.* 1034-45.—Heating 2.5 g. Mg, 13 g. $AcCH_2CO_2Et$, and 20 g. EtOH with a little CCl_4 initiator until the Mg dissolved, followed by addn. of 15 g. $ClCH_2CH:CHCH_2CH_2OMe$,

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g.). I with 1 mole PhLi gave in 3 hrs. (after CO_2 treatment) 24.4% II and 3.1% III; in 24 hrs. the amts. were 63% and 8.5%, while in 96 hrs. they were 78.4% and 7.0%, resp.

O. M. Kosolapoff

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analog (III). 9,10-Dihydro-1,2-benzanthracene gave, under a 1:1.1 molar relation, a mixt. of predominantly monocarboxylic acid, with about 3.3% of dicarboxylic acid admixt. Anthracene (1 g.), 0.2 g. Li, and 10 ml. Et_2O were shaken 140 hrs. and poured on solid CO_2, giving 90.0% *dicarboxylic acid*, m. 270-300°, after allowance for 10 mg. unreacted hydrocarbon. The above reaction mixt. treated with 1 g. I in Et_2O and let stand 40 hrs., then poured on CO_2, gave 2.04 g. II and 7.4% III; the mixt. m. 200-7°. To BuLi from 1.95 g. BuCl and 0.3 g. Li in 25 ml. Et_2O was added 3 g. I, the mixt. let stand in a closed ampul 20 hrs., and an ethylene oxide stream then passed into it; after aq. treatment, the org. layer was evapd., yielding, on soln. in a little 85% EtOH and standing, 0.36 g. I, while diln. of the mother liquor gave 0.9 g. *9-hydroxyethyl-9,10-dihydroanthracene*, m. 93-5° (from C_6H_6-petr. ether); chromatography of the residues on Al_2O_3 gave addnl. small amts. of this substance, as well as 2.5% *9,10-bis(hydroxyethyl)-9,10-dihydroanthracene*, m. 137-9° (from 50% EtOH). Treatment of BuLi (from 1.2 g. BuCl and 0.2 g. Li in Et_2O) with 2 g. 9,10-dihydro-1,2-benzanthracene 40 hrs. at room temp., followed by passage of MeCl, gave, upon treatment of the mixt. with picric acid, 0.13 g. *picrate* of *cis*(or *trans*)-*9,10-dimethyl-9,10-dihydro-1,2-benzanthracene*, m. 172-3°; the mother liquors, freed of picric acid, were dehydrogenated by S at 230-30° 0.5 hr. and chromatographed on Al_2O_3 in petr. ether; the products isolated included *10-methyl-1,2-benzanthracene*, isolated as the *picrate*, m. 171.5-3.5° (from C_6H_6) (0.18 g.), and *9-methyl-1,2-benzanthracene*, isolated as the *picrate*, m. 114-15° (0.41

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PROCESSES AND PROPERTIES INDEX

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Parachor and the Structure of Aromatic Amines (original text in Russian), B. A. Arbuzov and L. M. Guzhavina; J. Phys. Chem. (USSR) Sep '49 (23-9 Mthly); pp 1070-1074; 2 lb.

An investigation was conducted to determine the parachors of 14 aromatic amines. In order to compute the parachors of aromatic amines by the method of group values, the following group values were employed: $C_6H_5(N) = 189.4$; $(C)NH_2 = 44.5$; $(C)NH\text{-}(C) = 29.7$; $(C) - \underset{(C)}{N} - (C) = 13.0$; $(C) - C_6H_4(N) = 174.0$. The group value method for the determination of the theoretical values of amine parachors have very good corresponding values This method also enables one to show that in tertiary amines with long alkaline radicals per γ-carbonic atom chain, only two out of three chains are located parallel to each other. Corrections in the reactions of alkaline groups with nitrogen benzol chain were derived. With the exception of m-toluidine and η-octylaniline, the differences in the parachor values, derived by the group value method and by experiments, were not greater than 0.6-0.7%. On the basis of dioctyanaline and dicetyanaline parachors, it was shown that two alkaline chains of nitrogen with δ-carbonic atoms are located parallel to each other.

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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ARBUZOV, B. A.

USSR/Chemistry - Alcohols
Terpenes

May 49

"Activity of Alcohols on the Alpha-Oxide of Bicyclic Terpenes: I, Activity of Alcohols on the Oxide of Alpha-Pinene," Z. G. Isayeva, B. A. Arbuzov, Lab of Org Chem, Sci Res Inst imeni V. I. Ul'yanov-Lening, 8½ pp

"Zhur Obshch Khim" Vol XIX, No 5

Studies the activity of methyl and ethyl alcohols on the oxide of alpha-pinene in the presence of sulfuric acid, showing this reaction to be accompanied by isomerization of the oxide, mainly, into the corresponding methyl and ethyl acetals of campholene aldehyde. Interprets the isomerization under the effect of an acid, the activity of the alcohol with regard to the formation of "oxonium complexes" of pinene oxide with a hydrogen ion, the sobrerol, and the formation of campholene aldehyde during this last reaction. Submitted 7 Feb 48.

PA 67/49T52

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ARBUZOV, B. A.

USSR/Chemistry - Alcohols
Terpenes

May 49

"Activity of Alcohols on Alpha-Oxide of Bicyclic Terpenes: II, Activity of Alcohols on Oxides of Δ^3 - Carene and Camphene," Z. G. Isayeva, B. A. Arbuzov, Lab of Org Chem, Sci Res Inst imeni A. M. Butlerov, Kazan State U imeni V. I. Ul'yanov-Lenin, 12¼ pp

"Zhur Obshch Khim" Vol XIX, No 5

Studies the activity of primary, secondary, and tertiary alcohols on the oxide of Δ^3 -carene in the presence of sulfuric acid. From the primary are formed corresponding teritiary ethers of carene glycol. From isopropyl is formed, along with the isopropyl ether of carene glycol, unsaturated aldehyde through partial isomerization which gives in turn semicarbazole with a melting point of 216-218°. In the case of trimethylcarbinol, isomerization into the unsaturated aldehyde is the main reaction. Submitted 7 Feb 48

PA 67/49T51

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Action of alcohols on the α-oxides of bicyclic terpenes.
I. Action of alcohols on α-pinene oxide. Z. G. Isaeva and B. A. Arbuzov (Kazan State Univ.). *J. Gen. Chem. U.S.S.R.* 19, 871-80(1949)(English translation). II. Action of alcohols on Δ^3-carene and camphene oxides. — *Ibid.* 881-92. —See C.A. 44, 3166i. R. J. C.

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The parachor and structure of aromatic amines. B. A. Arbusov and L. M. Guzhavina. *Zhur. Fiz. Khim.* 23, 1070-4(1949); cf. *C.A.* 42, 8580i.—Surface tension γ was detd. at 20° by the max.-bubble-pressure method and the parachor P calcd. $PhNH_2$ had γ 43.66, P 234.0, PhNHMe 39.89 and 272.8, $PhNMe_2$ 36.46, 318.3; PhNEtMe 35.48, 348.3; $PhNEt_2$ 34.17, 386.0; *o*-toluidine 40.78, 270.8; *m*-toluidine 38.28, 269.6; *o*-$MeC_6H_4NEt_2$ 30.19, 422.6; *o*-$MeOC_6H_4NH_2$ 44.01, 289.6; PhNHPr (from $PhNH_2$, HCl, and PrOH at 180-200°, b_{11} 101-3°, n_D^{20} 1.5289, d_4^{20} 0.9404) 34.17, 347.6; PhNHBu (from $PhNH_2$ and *p*-$MeC_6H_4SO_3Bu$, b_{11} 111.5-12.5°, n_D^{20} 1.5330, d_4^{20} 0.9822) 33.76, 385.8; *n*-$C_6H_{17}NHPh$ (from $PhNH_2$ and $C_6H_{17}Br$ at 140-50°, b_{10} 162.5-3°, n_D^{20} 1.5130, d_4^{20} 0.9057) 32.90, 543.0; $(n\text{-}C_6H_{17})_2NPh$ (from *n*-$C_6H_{17}NHPh$ and $C_6H_{17}Br$ at 150-60°, b_{12} 207-8°, n_D^{20} 1.4998, d_4^{20} 0.8824) 31.7, 868.8. Dicetylaniline (from $PhNH_2$ and $C_{16}H_{33}I$ on water bath), m. 24°, was dissolved in PhH and its P calcd. (1481.1) from the γ of the soln. This P and those for PhNHEt and *p*-toluidine (literature data) agree with the values calcd. from the "group values" $C_6H_5(N)$ 189.4, $C(NH_2)$ 44.5, C(NH)-(C) 29.7, (C)-N(C)(C) 13.0, and (C)-C_6H_5-(N) 174.0 and several corrections of which the largest is for the parallel arrangement of the 2 chains in $(C_6H_{17})_2NPh$ (−25.2) and in dicetylaniline (−48.8). Only for *m*-toluidine and *n*-$C_6H_{17}NHPh$ are the calcd. Ps more than by 0.7% smaller than the exptl. Ps.

I. I. Bikerman

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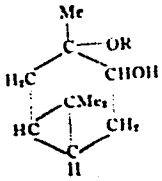
Action of alcohols on α-oxides of bicyclic terpenes. I. Action of alcohols on α-pinene oxide. Z. G. Isaeva and B. A. Arbuzov. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 19, 884-92(1949).—Addn. of MeOH or EtOH to α-pinene oxide in the presence of H_2SO_4 is accompanied by isomerization of the oxide, yielding Et or Me acetals of α-campholenaldehyde as the main products. The isomerization process is believed to proceed through the formation of oxonium type complexes of the oxide with the proton. α-Pinene oxide (from the oxidation of the hydrocarbon by AcO_2H) b_{12} 61–2°, n_D^{20} 1.4687, d_4^{20} 0.9056, $[\alpha]_D$ 67.58°, (58 g.) was added to 50 g. dry MeOH contg. 0.3 g. H_2SO_4 (spontaneous heating to 46°), let stand 3 hrs., neutralized with MeONa, and distd., giving a mixt. which could not be resolved completely; the entire mixt. had the range b_{12} 90–140°, from which was obtained about 13 g. product, b_{11} 96–101° (b_{14} 100–2° after distn. over Na), $C_{11}H_{22}O_2$, contg. 1 double bond and which on heating with 8% HCl 0.5 hr. at 60–8° gave the *semicarbazone*, m. 137–8° (from EtOAc), of *α-campholenaldehyde*. Treatment of the higher-boiling fractions with H_3BO_3 to remove OH derivs. gave an unstated amt. of the *campholenaldehyde di-Me acetal*, b_{14} 101–4°, n_D^{20} 1.4590, d_4^{20} 0.9392. In addn. the higher fractions gave about 17 g. product, $b_{11.5}$ 126–31°, n_D^{20} 1.4859, d_4^{20} 1.0000, $C_{11}H_{22}O_2$, contg. 10% OH groups (Zerewitinoff) and 1 double bond, which was provisionally given the structure of a *mono-Me ether* of an unsatd. glycol, probably *sobrerol*, at the tertiary C atom. Sobrerol is believed to form by HO ion addn. to the carbonium ion formed after protonic cleavage of the oxide ring. A similar reaction of 12 g. pinene oxide with 13 g. EtOH contg. 0.3 g. H_2SO_4 gave 9.4 g. of a mixt., b_6 90–114°, which on fractionation gave 4.1 g. $C_{11}H_{22}O_2$ (I), $b_{6.5}$ 88–9°, n_D^{20} 1.4646, d_4^{20} 0.9234, contg. 1 double bond, and giving *campholenaldehyde* on heating with 8% HCl, I thus being its *di-Et acetal*. II. **Action of alcohols on oxides of Δ³-carene and camphene.** *Ibid.* 893-905.—Δ³-Carene, b_{740} 167–8°, n_D^{20} 1.4720, d_4^{20} 0.8647, with AcO_2H gave the corresponding oxide, b_6 54–6°, n_D^{20} 1.4653, d_4^{20} 0.9516, $[\alpha]_D^{20}$ 14.03°; 31 g. added to 20 g. MeOH contg. 0.3 g. H_2SO_4 under 40°, followed by neutralization by NaOMe gave $C_{11}H_{22}O_2$, $b_{11.5}$ 112°, d_4^{20} 1.0065, n_D^{20} 1.4794, identified as the *carene glycol mono-Me ether* (I, R = Me); it could



(I)

be obtained in the pure state only after heating with H_3BO_3, followed by cleavage of the borate with hot water, and was obtained in but 8.6-g. yield. Similarly EtOH gave from 20 g. oxide 16.3 g. *Et ether* (I, R = Et), $b_{6.5}$ 93.5–4°, n_D^{20} 1.4758, d_4^{20} 0.9852, while PrOH gave 12.5 g. *Pr ether*, b_6 96–8°, n_D^{20} 1.4728, d_4^{20} 0.9692, and BuOH gave

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12, 694) that in general sulfinic acids add to certain quinones. *p*-AcNHC$_6$H$_4$SO$_2$H (19.9 g.) in 300 g. boiling H_2O was treated with 10.8 g. *p*-benzoquinone in 150 cc. warm H_2O in portions, set aside 20 min., cooled in ice, filtered, washed with H_2O (hot, then cold), and dried at 50°, giving 28 g. (91%) *2-(p-acetamidophenylsulfonyl)-hydroquinone* (I), m. 275-6° (decompn.) (from EtOH). I (20.7 g.) was refluxed 1 hr. in 117 cc. of 17% HCl, charcooled, cooled in ice, and the HCl salt (m. 228°) dissolved in 150 cc. hot H_2O plus 10 cc. concd. HCl, cooled in ice, and treated with solid Na_2CO_3 (phenolphthalein) to give 12.3 g. (50%) of the *amino compd.* (II), m. 178-9°. I (50 g.) in 70 cc. of 5 *N* NaOH dild. with 100 cc. H_2O was treated with 45 g. Me_2SO_4 (in 2 portions) with agitation, the mixt. warmed after 5 min. for 15 min. at 60° (steam bath), cooled, filtered, and the product washed with H_2O and dil. NaOH, giving 32.5 g. (60%) *2-(p-acetamidophenylsulfonyl)-1,4-dimethoxybenzene* (III), m. 214-15° (from MeOH). III (3.4 g.) in 5 cc. concd. HCl and 10 cc. EtOH was refluxed 1 hr., cooled, the HCl salt (IIIa) filtered, dissolved in 80 cc. hot H_2O and 10 cc. concd. HCl, and neutralized with concd. Na_2CO_3 soln. (litmus) to give 2.55 g. (85%) of the *amino compd.* (IV), m. 194-5°. IV (2.93 g.), 1.8 g. D-galactose, and 0.01 g. salicylic acid in anhyd. 30 cc. MeOH were refluxed 18 hrs., 15 cc. of the MeOH distd. off, and the mixt. set aside for 8 hrs., yielding 3.53 g. (77.5%) *2-(p-aminophenylsulfonyl)-1,4-dimethoxybenzene N-D-galactoside* (V) (dried over KOH), m. 198-9° (from EtOH). *p*-BzNHC$_6$H$_4$SO$_2$H (1.31 g.) in 50 cc. boiling 96% EtOH was treated with 0.55 g. *p*-benzoquinone in 19 cc. hot H_2O with stirring, and the mixt. poured after 15 min. in a thin stream with stirring into 1300 cc. cold H_2O, giving 1.8 g. (97%) *2-(p-benzamidophenylsulfonyl)hydroquinone* (VI), silky needles, m. 245-6°. II (6 g.) was added to 4.8 g. freshly distd. BzH in 10 cc. 96% EtOH, refluxed 40 min., most of the solvent evapd., the residue set aside, and the crystals filtered and purified with anhyd. ether, giving 5 g. (80%) *2-(p-benzylideneaminophenylsulfonyl)hydroquinone* (VII), m. 217-18° (decompn.), analyzed as IX. VII (1 g.) was treated with 0.295 g. $NaHSO_3$ in 4 cc. H_2O, heated on a steam bath 25 min., filtered, evapd. to a sirup on a steam bath, treated with 5 cc. of 100% EtOH, and set aside, giving after 2 hrs., 0.7 g. (54%) *Na 2-(p-benzylaminophenylsulfonyl)hydroquinone-α-sulfonate*, *p*-[2,5-(HO)$_2$C$_6$H$_3$SO$_2$C$_6$H$_4$N:CHPh]$_2$ (IX), which was filtered and washed (impn.), was prepd. in the same manner as VII from *2-(p-Anisylideneaminophenylsulfonyl)hydroquinone* (VIII) (64% yield), m. 206°. *Na 2-[p-(4-methoxybenzylamino)phenylsulfonyl]hydroquinone-α-sulfonate* (X) was prepd. in the same manner as IX (89%). *p*-Toluquinone (9 g.) in 20 cc. 96% EtOH was poured with stirring into 17.9 g. *p*-AcNHC$_6$H$_4$SO$_2$Cl in 280 cc. hot H_2O, the ppt. filtered, washed with hot H_2O, dried at 45°, and the product (XIIIA) (22 g., 93%) extd. with 50 cc. EtOH and filtered hot. The residue, after fractional crystn., yielded 1.9 g. *6(?)-(p-acetamidophenylsulfonyl)-p-toluquinone* (XIII), m. 272-3°. The hot alc. filtrate yielded, after fractional crystn., 1 g. *5(?)-(p-acetamidophenylsulfonyl)-p-toluquinone* (XI), m. 225-6° (*C.J.* 40, 180?). XI and XIII (4.5 g. XIIIA) in 20 cc. of 18% HCl-EtOH were refluxed 1 hr., the crystals filtered off after cooling, washed twice with 2.5 cc. of 18% HCl-EtOH, and the filtrate concd. to give a 2nd crop, yielding 2.3 g. XII.HCl, m. 218-19°. XII.HCl in cold dil. HCl (50 cc. H_2O plus some concd. HCl), carefully treated with Na_2CO_3, gave 1.3 g. *5(?)-(p-aminophenylsulfonyl)-p-toluquinone* (XII), m. 187-8° (from EtOH). The mother liquors of XII.HCl dild. with 10 vols. H_2O gave 1 g. *6(?)-(p-aminophenylsulfonyl)-p-toluquinone* (XIV), m. 247-8° (decompn.) (from EtOH). A sample obtained by the hydrolysis of XIII showed no m.-p. depression with XIV. XI was hydrolyzed to a product identical with XII. *p*-AcNHC$_6$H$_4$SO$_2$H (19.9 g.) in 200 cc. hot dil. (1:1) EtOH was treated with 16.4 g. thymoquinone in 50 cc. hot 80% EtOH with stirring; filtration, after chilling, and washing with dil. EtOH, gave 30 g. (83%) *3(?)-(p-acetamidophenylsulfonyl)thymohydroquinone*, 2,5,3,1,4-Me-

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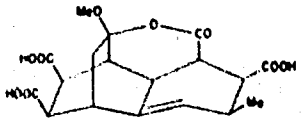
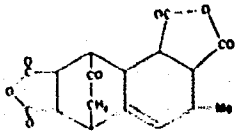
(no OMe group), m. 265-70° (decompn. began at 250°). VI, boiled a few hrs. with H_2O, gave IV in quant. yield; recrystn. from H_2O afforded the *tetrahydrate* (3 CO_2H and 1 lactone group) which again dried to IV. The colorless *dianhydride* (VIII), m. 298° (decompn. began at 275°)

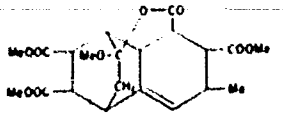


(VII)



(VIII)

(from $PhNO_2$), was prepd. by refluxing 2 g. IV a few min. in Ac_2O and washing the filtered product with Et_2O, or by heating IV near its m.p. Also, 2 g. VI, briefly boiled with 20 ml. HCO_2H, gave 1.1 g. VIII (no OMe group). All samples of VIII were identical (mixed m.p.) and that from IV gave back IV on hydrolysis. Treating 0.8 g. VII in 50 ml. MeOH with CH_2N_2 in Et_2O, concg. the soln. to 5 ml., and recrystg. the product twice from MeOH gave IX, m. 178-9° (distills undecompd. at 14 mm.), 0.3 g. of which, boiled a few min. in 1 ml. 98% HCO_2H, produced X, m. 230-2° (from MeOH), instantly sol. in aq. alkali. Treating 3 g. IV in 60 ml. MeOH with CH_2N_2 gave 2.5 g. (crude wt.) XI, which distills (14 mm.) undecompd. VI (20 g.), boiled 4 hrs. in 340 ml. MeOH and 40 ml. H_2SO_4, gave 20.5 g. (crude wt.) XI. With CH_2N_2, X also



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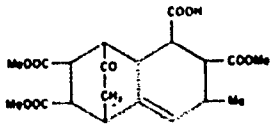
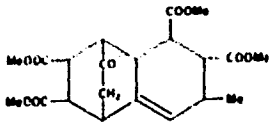


(X)



(XI)

gave XI. The 3 samples of recrystd. (from MeOH) XI were identical, m. and mixed m.p. 208-10°. VI (5 g.) was slowly dissolved in 100 ml. boiling 5 *N* HCl; concn. (reduced pressure) to 20 ml. gave 2.4 g. of the *trans-keto acid* (XII) (4 CO_2H groups), colorless needles, m. 200-2° (decompn.), after crystg. from H_2O and vacuum-drying at 100°; the *tetra-Me ester*, prepd. with CH_2N_2, formed colorless crystals, m. 154-5° (from MeOH). XI (1.7 g.) and IV (5 g.), refluxed 12 hrs. in 60 and 50 ml., resp., 5 *N* HCl, each gave (as did VII) XII, all samples of which were identical (mixed m.p.). Although XII (from IV) gave no large depression of the m.p. of IV, XII crystd. unchanged

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evapn. *in vacuo*, and treatment with NH_3 in Et_2O with cooling gave 6.7 g. IV amide. Heating 10 g. IV amide with excess $SOCl_2$ 8 hrs. under reflux, evapn. *in vacuo*, and extn. by Et_2O gave: 0.5 g. β-phenyl-β-alanine, 0.1 g. cinnamic acid, 0.8 g. of a substance m. 82-3° (from EtOH), apparently $HO_2CCH_2CHPhNAcCO_2Et$, and 6.75 g. oil, sepd. by distn. into PhCH:CHCONHAc, b. 195-200°, and a fraction b. 120-95°, which, warmed with 5% NaOH, gave cinnamic acid and IV; hence the oil is probably 2-ethoxy-4-phenyl-6-oxo-3,4,5,6-tetrahydropyrimidine.

G. M. Kosolapoff

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ARBUZOV, B.A.

USSR/Chemistry - Diene, Synthesis
Chemistry - Synthesis

Apr. 49

"Diene Synthesis With Selenophene and Its Homologues," B. A. Arbuzov, Corr Mem, Acad Sci USSR, Ye. G. Katayev, Sci Res Chem Inst imeni A. M. Butlerov, Kazan State U imeni V. I. Ul'yanov-Lenin, 3 pp

"Dok Ak Nauk SSSR" Vol LXV, No 4

Showed that selenophone, 2-methylselenophene, and 2,5-dimethylselenophene enter into diene synthesis with maleic anhydride during heating at 150° for 16 hours. Diene synthesis does not occur at

41/49T1

USSR/Chemistry - Diene, Synthesis (Contd) Apr 49

lower temperatures (100 - 120°). Separation of elementary selenium was observed in all reactions, but separation of hydrogen selenide was not observed. Submitted 3 Feb 49.

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Dipole moments of esters of orthocarbonic acids. B. A. Arbuzov and T. G. Shavaha. *Doklady Akad. Nauk S.S.S.R.* 68, 515-17(1949).—From dielec.-const. measurements in dil. solns. in C_6H_6 at 20 and 25°, with the electronic polarization taken equal to the mol. refraction, the dipole moments of the following tetraalkyl esters of $C(OH)_4$ were detd. Me 0.83, Et 1.1, Bu 1.00, hexyl 1.00, octyl 0.98, nonyl 1.17, decyl 1.06 D. The value for the Me ester is close to that of Ebert and Hartel (*C.A.* 21, 3408) but at variance with Fuchs (*C.A.* 24, 5551). All exptl. values are close to an av. 1.1, and considerably below the 1.70 D., calcd. by Zahn's formula (*C.A.* 26, 4219) under the assumption of free rotation of the OR groups.

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CA

Dipole moments of esters of orthosilicic and of orthotitanic acids. B. A. Arbuzov and T. G. Shavtsha. *Doklady Akad. Nauk S.S.S.R.* 68, 859-60(1949).—(1) From measurements of the dielec. const. in C_6H_6 soln. at 20°, with P_{e+a} (electronic + at. polarization) = 1.05 MR_D, the dipole moments of esters of $Si(OH)_4$ were detd.: Me ester 1.61, Et 1.70, Pr 1.66, Bu 1.65, C_5H_{11} 1.66, C_7H_{15} 1.64, C_8H_{17} 1.60, C_9H_{19} 1.75, $C_{10}H_{21}$ 1.63 D. For esters of $Ti(OH)_4$, the exptl. values are: Bu 1.08, C_5H_{11} 1.62, C_7H_{15} 1.60, C_8H_{17} 1.67, C_9H_{19} 1.08 D. In both cases, the values are close to 1.70, calcd. on the assumption of symmetrically tetrahedral grouping around the Si or Ti atom, and unhindered rotation of the O—R groups. This agreement contrasts with the big difference between the exptl. and the calcd. moments of esters of orthocarbonic acid (A. and S., *C.A.* 44, 392*e*). (2) The esters of $Ti(OH)_4$ were newly synthesized by exchange with $Ti(OEt)_4$ and had the following consts.: Bu, $b_{0.4}$ 166°, d_4^{20} 1.0051, n_D^{20} 1.4925; C_5H_{11}, b_5 201-4°, 0.9573, 1.4830; C_7H_{15}, b_1 232-5°, 0.8963, 1.4610; C_8H_{17}, b_3 255-8°, 0.9339, 1.4810; C_9H_{19}, $b_{1.4}$ 264-5°, 0.9241, 1.4785. N. Thon

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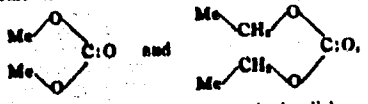
Dipole moments of cyclic esters of carbonic acid. B. A. Arbuzov and T. G. Bhavsha. *Doklady Akad. Nauk S.S.S.R.* 69, 1045-8(1949).—In connection with G. Thomson's calcns. (*C.A.* 33, 8461[1]) of dipole moments for the 2 possible configurations of Me_2CO_3 and the 3 configurations of Et_2CO_3, leading to the conclusion of the nonoccurrence of the configurations



and

detns. of the dipole moments were made, by dielec. const. measurements in C_6H_6 soln., for 3 cyclic esters close to the above improbable configurations of Me_2CO_3 and Et_2CO_3. Exptl. values are O.CO.O.CH_2.CH_2 4.80, O.CO.O.CH_2.-CH_2.CH_2 5.21, O.CO.O.CHMe.CH_2.CH_2 5.98 D. The excess of these values over those calcd. by Thomson for the improbable configurations is due to ring closure, and the corresponding increment, 0.6 Debye, checks with that found by Robles (*C.A.* 33, 2785[8]) for the dipole-moment difference between simple alkyl and polymethylene oxides. Six-membered rings have a dipole moment about 0.3–0.4 greater than five-membered rings, owing to nonplanar configuration. N. Thou

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Dipole moments of cyclic esters of sulfurous acid. II. A. Arbuzov and T. G. Shavshin. *Doklady Akad. Nauk S.S.S.R.* 30, 41-3(1940).—The exptl. value of the dipole moment of diethyl sulfoxylate, $S(OEt)_2$, μ = 1.90 D., is fairly close to μ = 1.73 calcd. with the angle O—S—O = 103°, the bond moments S—O = 1 and O—Et = 1.12, and on the assumption of free rotation of the OEt groups around S—O; it is intermediate between 0.90 and 2.42, calcd. for the 2 extreme rigid structures. From dielec. const. measurements in C_6H_6 soln., $O.CH_2.CH_2.O.S:O$ (I) has μ = 3.65, $O.CH_2.CH_2.CH_2.O.S:O$ (II) μ = 3.00 D. For I, calcn. with the above angle (103°) and bond moments (S—O = 1, O—CH_2 = 1.12) and S—O = 3.0 D., on the assumption of a plane structure of the ring, gives μ = 3.46; this, plus an increment of 0.3 for removal of 2 H atoms and ring closure (on the basis of the difference of μ Et_2S and $CH_2.CH_2.CH_2.CH_2.S$) gives μ = 3.76, close to the exptl. 3.65. The closeness of the μ of I and II can be due either to nonplanar structure of the ring in II alone, or in both II and I. The value of 3.00 is the mean of 5.21 and 1.39, calcd. for the 2 possible nonplanar configurations, plus 0.3 for ring closure.

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Parachors of cyclic acetals. B. A. Arbuzov and V. S. Vinogradova (Kazan State Univ.). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1950, 201-6; cf. *Doklady Akad. Nauk S.S.S.R.* 59, 65(1947); *C.A.* 42, 5735*e*, 5844*h*, 8560*d*.—The parachor of 1,3-dioxane (I) was detd. experimentally to be 303.21, that of dioxolane $-OCH_2OCH_2CH_2$ (II) to be 168.1. The difference, 37.0, is very close to the 36.7 calcd. on the assumption that passage from a six-membered to a five-membered ring should decrease the standard group parachor of CH_2, 42.0, by 5.3. Consequently, the ring correction valid for carbocyclic compds. is applicable also to oxygenated heterocycles. The parachors calcd. from the ideal group parachors, with the corresponding ring corrections (10.7 and 5.4 for the six- and the five-membered ring, resp.) are 311.11 and 174.25, for I and II, resp.; the deviation, av. 6.0, is attributed to the O atoms, and leads to the introduction of a correction of −3.0 for each O atom in the heterocyclic ring. With this correction, and the previously established corrections for the interaction between the side chain and the ring, and between the side chains, the calcd. parachors are in very good agreement (deviation not exceeding 0.4%) with the following experimentally detd. parachors: 4-methyl-1,3-dioxane 344.6; 2-methyl-1,3-dioxane 343.3; 2-methylidioxalane 207.0; 2-phenylidioxalane 342.3; 2,4-dimethyl-1,3-dioxane 382.5; 1,4-dioxane 305.4; 2-phenyl-1,3-dioxane 378.7. Cf. following abstr. N. Thon

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No. 3

Parachors of the cyclic esters of carbonic, sulfurous, and phosphorous acid. B. A. Arbuzov and V. S. Vinogradova (Kazan State Univ.). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1950, 297-303; cf. preceding abstr.—The exptl. value of the parachor of ethylene glycol carbonate $CH_2OCOOCH_2$, 179.0, is in agreement (within 0.5%) with the 179.9 calcd. with 107.0 taken for the ideal group parachor of (C)OCOO(C); the latter value is obtained from the group parachor, 85.5, with the corrections −5.2 for the interaction of atoms in O<(C)(C), −3.7 in C<(O)(O), and −3.7 in C<(O)(O). Further corrections applied are −5.4 for the five-membered ring, and −6.0 for the 2 O atoms in the ring (cf. preceding abstr.). By the same procedure, the parachor of trimethylene glycol carbonate $CH_2CH_2OCOOCH_2$ is calcd. to be 216.7, within 0.5% of the exptl. 217.8, and that of $CH_2CH_2OCOOCHMe$ is calcd. to 254.0, within 0.2% of the exptl. 254.4. For the group (C)OSOO(C), the ideal group parachor is estd. to be 121.5, which, with the usual corrections for the ring and the 2 O atoms, fits the parachors of $CH_2OSOOCH_2$ (calcd. 194.4, exptl. 194.3) and $CH_2CH_2OSOOCH_2$ (calcd. and exptl. 231.3). In an analogous way, the ideal group parachor of $[(C)O]_2P$ in five- and 6-membered rings is calcd. to be 109.7; with this value, and the usual corrections, the calcd. parachors agree within better than 0.8% with the exptl. values: $CH_2OP(OMe)OCH_2$ 244.00, $CH_2OP(OEt)OCH_2$ 281.4, $CH_2CH_2OP(OMe)OCHMe$ 319.6, $CH_2CH_2OP(OEt)OCHMe$ 353.4.

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Diene syntheses with piperylene. B. A. Arbuzov and E. G. Kataev (Kazan State Univ.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 68-81(1950).—Piperylene (I) (20 g.), 16.4 g. CH_2:CHCN, and 0.1 g. hydroquinone after 24 hrs. at 130-40° gave 24 g. (64%) *2-methyl-3-cyclohexene-1-carbonitrile*, b_{15} 69-70°, b_{15} 73-4°, n_D^{20} 1.4683, d_4^{20} 0.9289; this in boiling abs. EtOH in a dry HCl stream gave after 12 hrs. 40% of the corresponding *Et ester*, b_{15} 95-7°, b_{100} 138-40°, n_D^{20} 1.4619, d_4^{20} 0.9741, which on boiling with aq. alc. Na_2CO_3 12 hrs. gave 73% of the *free acid* (II), sirup, b_{20} 138-40°, n_D^{20} 1.4738, d_4^{20} 1.0397: *Ag salt*, colorless powder. Ozonization of 5 g. free acid for 16 hrs. in 50 ml. H_2O contg. 3 g. Na_2CO_3, followed by addn., in the cold, of 800 ml. 1% $KMnO_4$ soln., gave *1,3,4-pentanetricarboxylic acid*, m. 176-8° (from HCl). The above nitrile (1.3 g.) was dehydrogenated by heating 2 hrs. to 200° with 3.41 g. Br, then boiling 16 hrs. with 30% NaOH, which gave *o*-toluic acid, confirming the structure. Heating 60 g. acrolein, 115 g. I, and 0.5 g. hydroquinone 8 hrs. to 125-30° gave 70 g. (56%) *2-methyl-3-cyclohexen-1-al*, b_{15} 56-8°, n_D^{20} 1.4653, d_4^{20} 0.9435 [*semicarbazone*, m. 167-8° (from EtOH)]; oxidation of the aldehyde (25 g.) with 65 g. $AgNO_3$ and 30 g. NaOH in dil. EtOH over 12 hrs. gave the trans form of II, m. 52-2.5° (from petr. ether); its *Ag salt* is a colorless powder; transformation to the acid chloride (by PCl_5) and treatment with NH_4OH gave the *amide*, m. 157-8° (from H_2O); the liquid form of II failed to give the amide. I (18 g.) and 25 g. MeCH:C(CO_2Et)$_2$ in the presence of hydroquinone gave after 13 hrs. at 170-5° 74% *di-Et 2,6-dimethyl-3-cyclohexene-1,1-dicarboxylate*, b_{15} 123-4°, b_{11} 130-8°, n_D^{20} 1.4619, d_4^{20} 1.0416; hydrolysis with 25% EtOH-KOH 8 hrs. at 90° gave the *free acid*, m. 188° (from EtOAc), in 42% yield; decarboxylation at 200° gave 70% of the *monocarboxylic acid*, m. 55-6° (from EtOAc), which, heated 24 hrs. at 360° with Se, then oxidized with $KMnO_4$, gave isophthalic acid. I (14 g.) and 25 g. PhCH:C(CO_2Et)$_2$, in the presence of hydroquinone, gave in 12 hrs. at 190° 39.3% *di-Et 2-methyl-6-phenyl-3-cyclohexene-1,1-dicarboxylate*, b_{15} 170-2°, n_D^{20} 1.5206, d_4^{20} 1.1006; heating this with 25% alc. KOH 12 hrs. gave the *free acid* (54%), m. 183° (decompn.; from EtOAc), which on heating to 190° gave a sirupy *monocarboxylic acid* (isolated as the *Ag salt*), while dehydrogenation with Se at 360° and oxidation of the product with $KMnO_4$ gave *3-phenylbenzoic acid*, m. 159-61° (from dil. EtOH). I (14 g.) and 16 g. AcC(:CHMe)CO_2Et similarly gave after 18 hrs. at 180° 74% *Et 2,6-dimethyl-1-acetyl-3-cyclohexene-1-carboxylate*, b_{15} 116-18°, n_D^{20} 1.4724, d_4^{20} 1.0326, which on sapon. with alc. KOH gave 45% *2,6-dimethyl-1-acetyl-3-cyclohexene*, b_{15} 80-1°, n_D^{20} 1.4635, d_4^{20} 0.9259 (*semicarbazone*, m. 124°), and an unstated amt. of *2,6-dimethyl-3-cyclohexene-1-carboxylic acid*, m. 56°. Similar reaction of I with AcC(:CHPh)CO_2Et at 180° for 20 hrs. gave 44.5% *Et 2-methyl-6-phenyl-1-acetyl-3-cyclohexene-1-carboxylate*, b_{15} 165-7°, n_D^{20} 1.5288, d_4^{20} 1.0980, and 20.5% *Et 3-methyl-2-phenyl-1-acetyl-4-cyclohexene-1-*

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carboxylate, b_4 107-71°, m. 61° (from MeOH). Hydrolysis of the former ester with 25% alc. KOH gave 27% *2-methyl-6-phenyl-1-acetyl-3-cyclohexene*, b_{10} 150-6°, n_D^{20} 1.5301, and 53% *2-methyl-6-phenyl-3-cyclohexene-1-carboxylic acid*, sirup, b_4 160-70° (isolated as the Ag salt); dehydrogenation with Se as above gave *m*-$PhC_6H_4CO_2H$). Hydrolysis of the latter ester with alc. KOH gave 26% *3-methyl-2-phenyl-1-acetyl-4-cyclohexene* (structure uncertain; gives a semicarbazone, m. 195°), and 70% *3-methyl-2-phenyl-4-cyclohexene-1-carboxylic acid*, m. 83° (from dil. MeOH), which on dehydrogenation with Se and treatment with $KMnO_4$ gave *2-phenylbenzoic acid*, m. 112-13°.

G. M. Kosolapoff

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Organophosphorus-tin compounds. III. Synthesis of compounds with phenyl radicals at the phosphorus atom. B. A. Arbuzov and N. P. Grechkin (Chem. Inst., Kazan Div. Acad. Sci. U.S.S.R.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 107-15(1950); cf. *C.A.* 42, 4522a. — $PhP(OMe)_2$ (2.8 g.) and 4.7 g. Me_3SnI warmed to 80° reacted violently (max. temp. 240°) and yielded 3.4 g. *PhPO(OMe)(SnMe₃)*, m. 132-4° (from EtOH). Similarly, 6.4 g. $PhP(OEt)_2$ with 10.8 g. Et_3SnI preheated to 150° gave 1.6 g. insol. colorless *PhPO(OEt)(SnEt₃)*, m. 204-7°. $PhP(OEt)_2$ (4.3 g.) and 9.5 g. Bu_3SnI at 185° gave 7.2% insol. *PhPO(OEt)(SnBu₃)*, m. 302-5° (decompn.), although 80% EtI was evolved. Heating $PhPO(OMe)(SnMe_3)$ 30 min. with H_2O gave no change, but 15% HCl at room temp. yields Me_3SnCl rapidly, while 10% KOH in 3 hrs. yields Me_3SnOH, m. 117-20°. $PhP(OMe)_2$ (5.1 g.) and 6 g. Me_3SnI gave after a violent reaction 3.7 g. *$Me_3Sn[P(O)(OMe)Ph]_2$*, decomp. 201-3°. Similarly 7 g. Et_3SnI and 6.5 g. $PhP(OEt)_2$ after preheating to 150° gave 2.8 g. *$Et_3Sn[P(O)(OEt)Ph]_2$*, decompg. 280-300°, while 4.5 g. Et_3SnI and 5 g. $PhP(OPr)_2$ gave, at 195°, 61.4% *$Et_3Sn[P(O)(OPr)Ph]_2$*, decompg. 246-8°. All these products are insol. in org. solvents; they are stable to hot H_2O, but rapidly cleave in 15% HCl, yielding R_3SnCl_2, while 10% NaOH yields R_3SnO; Cl in $CHCl_3$ also yields R_3SnCl_2 at room temp. Heating $PhPCl_2$ 8 hrs. in a sealed tube to 300° gave 30% *Ph_2PCl*; this with MeOH in the presence of Me_3NPh gave 55% *Ph_2POMe*, b_{10} 152-5°, while EtOH gave 51% *Ph_2POEt*, b_{10} 161-3°. Heating 7 g. Et_3SnI and 4.9 g. Ph_2POEt to 170° gave 5.1% *$Ph_2P(O)SnEt_3$*, decompg. 346-8°, while the EtOH-Et_2O washings gave 1.4 g. *Ph_2EtPO*, m. 121-4°. Likewise, 5.1 g. Me_3SnI and 4 g. Ph_2POEt gave 9.3% *$Ph_2P(O)SnMe_3$*, decompg. 365-8°, and 1.75 g. Ph_2EtPO, while 4.6 g. Et_3SnI heated with 3 g. Ph_2POMe to 190° gave *Ph_2MePO*, m. 108-10°, and 4.2 g. (91.5%) *Et_3SnI*. Me_3SnI (6 g.) and 6.9 g. Ph_2POEt heated to 140° gave 2.2 g. *$Me_3Sn[P(O)Ph_2]_3$*, decompg. 372-5°, and 0.9 g. Ph_2EtPO, while 5.6 g. Et_3SnI and 6 g. Ph_2POEt gave 17.3% *$Et_3Sn[P(O)Ph_2]_3$*, decomp. 351-3°, and Ph_2EtPO. These derivs. are stable to hot H_2O, while warm 15% HCl yields Ph_2PO_2H, m. 193-4°, and R_3SnCl_2, and hot 20% NaOH similarly gives Ph_2PO_2H and R_3SnO; Cl also yields R_3SnCl_2 and presumably Ph_2POCl, for aq. treatment with water gives Ph_2PO_2H; the action of AcCl in refluxing C_6H_6 is similar, as R_3SnCl_2 and Ph_2PO_2H are isolated.

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Diene syntheses with 2,4-hexadiene. B. A. Arbuzov and B. G. Kataev (Kazan State Univ.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 931-8(1950); cf. *C.A.* 44, 8027c. *2,4-Hexadiene* (I) enters the diene synthesis less readily than piperylene. I (30 g.), 18 g. $CH_2:CHCN$, and 0.1 g. hydroquinone after 12 hrs. at 120-30° in a sealed tube gave 40% *2,5-dimethyl-3-cyclohexene-1-carbonitrile*, b_8 82-3°, n_D^{20} 1.4678, d_4^{20} 0.9202; heated with the calcd. amt. of Br 2 hrs. at 200°, then refluxed 18 hrs. with aq. alc. NaOH, it gave 2,5-$Me_2C_6H_9CO_2H$, m. 125° (17 g.), 25 g. $MeCH:C(CO_2Et)_2$, and hydroquinone (0.2 g.) after 13 hrs. at 170-5° gave 31% *di-Et 2,5,6-trimethyl-3-cyclohexene-1,1-dicarboxylate*, b_5 128-9°, b_{10} 144-6°, n_D^{20} 1.4641, d_4^{20} 1.0362, which on heating 7 hrs. with aq. alc. KOH gave *2,5,6-trimethyl-3-cyclohexene-1-carboxylic acid*, b_4 160-8°, an oil [amide, m. 183-4° (from MeOH)]. Similarly, I with $MeCH:C(CO_2Et)Ac$ for 18 hrs. at 180° gave 40% *Et 2,5,6-trimethyl-1-acetyl-3-cyclohexene-1-carboxylate*, b_4 115-17°, n_D^{20} 1.4744, d_4^{20} 1.0247, which on heating with 25% aq. alc. NaOH gave 55% *2,5,6-trimethyl-1-acetyl-3-cyclohexene*, b_{10} 115-18°, n_D^{20} 1.4651; *semicarbazone*, m. 165-7° (from MeOH). I with $PhCH:C(CO_2Et)_2$ gave in 12 hrs. at 180° 30% *di-Et 2,5-dimethyl-6-phenyl-3-cyclohexene-1,1-dicarboxylate*, b_4 170-80°, n_D^{20} 1.5231, d_4^{20} 1.0945. $PhCH:C(CO_2Et)Ac$ gave 40% *Et 2,5-dimethyl-6-phenyl-1-acetyl-3-cyclohexene-1-carboxylate*, b_4 163-5°, n_D^{20} 1.5300, d_4^{20} 1.0806.

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Synthesis of esters of naphthylmethanephosphonic acids

B. A. Arbuzov and B. P. Lugovkin (A. M. Butlerov Chem. Inst., Kazan Univ.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) **20**, 1249-51(1950); cf. *C.A.* **42**, 7265g; Kosolapoff, *C.A.* **40**, 1443^3.—Addn. of 2.0 g. $(EtO)_3P$ over 15 min. to 3 g. 2-$C_{10}H_7CH_2Br$, m. 52.3° [Schulze, *Ber.* **17**, 1520(1884)], kept at 140-50°, and heating to 170-80° gave 87.3% 2-$C_{10}H_7(CH_2PO)(OEt)_2$, b. 170-2°, n_D^{20} 1.5583, d_4^{22} 1.1483, which heated 5 hrs. to 125-35° in 1:1 HCl in a sealed tube gave 98.0% *free acid*, m. 229-30° (from H_2O). Addn. of 3.5 g. $(EtO)_3P$ to 1.7 g. 1,4-$C_6H_4(CH_2Cl)_2$, m. 143-5° (Badger, *et al.*, *C.A.* **42**, 2249*h*), at 150-5°, followed by heating 1 hr. at 200-5°, gave 3.2 g. undistillable, viscous 1,4-$C_6H_4(CH_2PO(Et)_2)_2$, which hydrolyzed as above 8 hrs. at 130-5° gave 93.7% *free diphosphonic acid*, m. 275-8° (crude), m. 276-9° (from H_2O). Similarly, 1.2 g. $(EtO)_3P$ and 2.4 g. 5,8-bis(chloromethyl)-1,2,3,4-tetrahydronaphthalene gave 66.3% *tetra-Et 5,8-bis(phosphonomethyl)-1,2,3,4-tetrahydronaphthalene*, b. 254-5°, n_D^{20} 1.5146, which on hydrolysis, as above, gave 93.7% *free acid*, needles, m. 290-300° (from H_2O at 135°). The intermediate chloromethyl deriv., m. 117°, was prepd. according to Badger, *et al* (cf. above).

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The synthesis of esters of naphthylmethanephosphonic acids. B. A. Arbuzov and B. P. Lugovkin. *J. Gen. Chem. U.S.S.R.* 20, 1297-9(1950)(Engl. translation).—See *C.A.* 45, 1567*a*. R. M. S.

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ARBUZOV, B. A.

"The action of a- and y-bromacetoacetic ester and of 2-chlorocyclohexan-one on trietlyl phosphite and on the sodium compound of dietlyl phosphite." B. A. Arbuzov, B. P. Lugovkin, and N. P. Bogonostseva. (p. 1468)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii) 1950, Vol 20, No 8.

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The action of α- and γ-bromoacetoacetic esters and of 2-chlorocyclohexanone on triethyl phosphite and on sodium diethyl phosphite. B. A. Arbuzov, B. P. Lugovkin, and N. P. Bogomostseva. *J. Gen. Chem. U.S.S.R.* 20, 1529-37(1950)(Engl. translation).—See *C.A.* 45, 1506a.

R. M. S.

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Parachor and structure of esters of orthopropionic acid. B. A. Arbuzov and E. K. Yuldasheva (Kazan. Gosudarst. Univ. im. V. I. Ul'yanova-Lenina). *Doklady Akad. Nauk S.S.S.R.* 70, 231-2(1950).—(1) Experimentally detd. parachors of esters $EtC(OR)_3$ are: R = Et, 436.4; iPr, 549.3; Bu, 669.9; C_6H_{11}, 905.8; C_8H_{17}, 1145.4. Comparison of the exptl. values with those calcd., for R = C_6H_{11} and C_8H_{17}, on the 2 alternative assumptions of an interaction between all 3 or only between 2 of the R groups, has decided in favor of the latter alternative. (2) The new esters were synthesized by exchange between $EtC(OEt)_3$ (I) and the corresponding alc. ROH, in 30-40% excess over the theory, in the presence of H_3PO_4 (d. 1.8), 0.5 ml./0.1 mole I; the mixt. is heated until no more EtOH is evolved. The new compds. have the consts.: $EtC(OiPr)_3$ b_{14} 92.5-3°, d_4^{20} 0.8874, γ^{20} 24.55, n_D^{20} 1.41227; $EtC(OBu)_3$ b_4 117-17.5°, 0.8749, 25.67, 1.42321; $EtC(OC_6H_{11})_3$ b_4 172.5-3.5°, 0.8699, 27.35, 1.43500; $EtC(OC_8H_{17})_3$ $b_{0.4}$ 207-8°, 0.8664, 28.85, 1.44205. N. Thon

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Parachors and structure of dialkylphosphorous acids. B. A. Arbuzov and V. S. Vinogradova (V. I. Ul'yanov-Lenin State Univ., Kazan, U.S.S.R.). *Doklady Akad. Nauk S.S.S.R.* **72**, 695-8(1950). In the series $(C_nH_{2n+1}O)_2POH$, n = 1-8, the mean increment, Δ, of the parachor per CH_2 group is 39.6, close to the normal value of 39.8. The higher members have Δ = 40.2, which corresponds to parachors P of the order of 400-700, whereas if these higher members were dimerized, one should expect P = 1200-1500 and Δ = 40.8-41.1. Absence of assocn. in $(C_4H_{10}O)_2POH$ was confirmed by cryoscopy in C_6H_6. In contrast thereto, $(MeO)_2POH$ is assocd. and is monomeric only in very dil. soln. From the exptl. P = 764.0 of the authentically monomeric $(C_4H_{10}O)_2POH$, the group parachor of $[(C)O]_2P\langle^{O}_{H}$, corrected for expansion, bending, and interaction of the chains, is 107.3. Comparison of the P values calcd. with this new group parachor, with the exptl. P, shows that only the members with n = 8, 7, and 6, are monomeric. For the lower members, deviations between the calcd. and the exptl. P indicate assocn., increasing with decreasing n. On the other hand, if the group parachor $[(C)O]_2P\langle^{O\cdots H}_{H\cdots O}\rangle P[O(C)]_2$ is calcd. on the assumption of complete dimerization of $[(MeO)_2POH]_2$, it is found = 229.8, and then comparison of the calcd. and the exptl. P indicates increasing dissocn. of the dimer to monomers with increasing n. The per cent deviation becomes const., −1.7%, from n = 6 up, which confirms that the members with n = 6, 7, and 8, are completely monomeric. On that basis, the percentage of monomer at 20° can be roughly estd. to be 65% in $(iso\text{-}BuO)_2POH$, 70% in $(BuO)_2POH$, and 40% in $(PrO)_2POH$. The exptl. values of P for the $(RO)_2POH$ are: R = Me, 452.65; Et, 605.3; Pr, 758.0; iso-Pr, 755.6; Bu, 909.4; iso-Bu, 908.2; C_6H_{13}, 1216.0; C_7H_{15}, 1372.0; C_8H_{17}, 1528.0.

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No. 2

Addition of dialkyl phosphites to unsaturated ketones, nitriles, and esters. A. N. Pudovik and B. A. Arbuzov (A. E. Arbuzov Inst., State Univ., Kazan). *Doklady Akad. Nauk S.S.S.R.* 73, 327-30(1950); cf. *C.A.* 44, 1893g.—The addn. of $(RO)_2PONa$ to unsatd. compds., discovered earlier (cf. above ref.) in reactions of secondary allylic chlorides, was extended to other unsatd. compds. with an active unsatd. link; compds. contg. CO, CN, or CO_2R groups which by their electron-attractive properties produce a pos. charge on the β-C atom of the ethylene link aid the addn. of nucleophilic reagents to the unsatd. link, as the addn. to the carbonyl group itself is correspondingly repressed. These unsatd. derivs. readily add $(RO)_2POH$ in the presence of alcoholates of alkali metals, the reaction being run by dropwise addn. of a satd. alc. soln. of RONa to an equimolar mixt. of $(RO)_2POH$ and the unsatd. deriv. The action is exothermic (the effect is smaller with larger compds.) and the products are isolated by distn. after an unstated period required for completion. Usually the phosphites of the higher alcs. require larger amts. of RONa for satisfactory action; it is advisable to use the same R in RONa and the phosphite to avoid transesterifications. $Me_2C:CHCOCH:CH_2$ yields products contg. 1 double bond (Br titration), while hydrolysis of the product obtained with $(EtO)_2POH$ by means of HCl yielded EtCl (in an amt. corresponding to an equimolar adduct); no H_3PO_4 was detected (H_3PO_4 would form if the phosphono group attached itself to the α-C atom in respect to the CO group), and the product showed an active keto group by qual. reactions with Na nitroprusside and $2\text{-}C_{10}H_7OH$, and by formation of a phenylhydrazone. On this basis the structures of the following products, $Me_2C:CHCOCH_2CH_2PO(OR)_2$, were established (R given): *Me* (59%), b_{10} 169-71°, n_D^{20} 1.4711, d_4^{20} 1.1130; *Et* (52%), b_5 149°, d_4^{20} 1.0658, n_D^{20} 1.4600; *iso-Bu* (48.5%), b_{10} 188-9°, n_D^{20} 1.4511, d_4^{20} 1.0013; *Bu* (57%) b_{10} 198-200°, n_D^{20} 1.4623, d_4^{20} 1.0111(?). The addns. to $CH_2:CHCN$ are much more energetic and addn. of a few drops of RONa soln. to the mixt. causes spontaneous boiling, unless the mixt. is cooled; hydrolysis of the adducts gave phosphonopropionic acid, m. 164-5°, thus confirming the structure $(RO)_2P(O)CH_2CH_2CN$: R = *Me* (86.3%), b_{10} 158°, n_D^{20} 1.4432, d_4^{20} 1.1064; *Et* (83%), b_{10} 159-60°, n_D^{20} 1.4388, d_4^{20} 1.1089; *iso-Pr* (46.4%), b_{10} 160°, n_D^{20} 1.4345, d_4^{20} 1.0500; *iso-Bu* (71%), b_{10} 171°, n_D^{20} 1.4386, d_4^{20} 1.0359; *Bu* (71.5%), b_{10} 177-8°, n_D^{20} 1.4395, d_4^{20} 1.0155. With $CH_2:CMeCO_2Me$ the reaction is much more sluggish and the action begins only after addn. of considerable amts. of RONa; the products obtained, $(RO)_2P(O)CHMeCO_2Me$, were: R = *Me* (77.7%), b_{10} 137-8°, n_D^{20} 1.4377, d_4^{20} 1.1761; *Et* (74.5%), b_{10} 152-3°, n_D^{20} 1.4350, d_4^{20} 1.1212; *iso-Pr* (61%), b_{10} 153-4°, n_D^{20} 1.4298, d_4^{20} 1.0612. The reactions appear to proceed by addn. of $(RO)_2PO^-$ and Na^+ to the unsatd. link, with subsequent replacement of Na by H supplied by a 2nd mole of $(RO)_2POH$, thus regenerating $(RO)_2PONa$.

G. M. Kosolapoff

TRANSLATION AVAILABLE (U-1126)

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ARBUZOV, B. A.

PUDOVIK, A.N.; ARBUZOV, B.A.

Addition of dialkylphosphorous acids to unsaturated compounds. Part 1: Addition of dialkylphosphorous acids to β, β-dimethyldivinylketone. Izv.Kazan.fil.AN SSSR Ser.khim.nauk no.1:45-52 '50. (MLRA 10:5)

(Phosphorous acids) (Vinyl compounds)

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ARBUZOV, B.A.

PUDOVIK, A.N.; ARBUZOV, B.A.

Addition of dialkylphosphorous acids to unsaturated compounds. Part 2: Addition of dialkylphosphorous acids to the nitrile of acrylic acid and methylmethacrylate. Izv.Kazan.fil.AN SSSR Ser. khim.nauk no.1:53-58 '50. (MLRA 10:5)

(Phosphorous acids) (Methacrylic acid)

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ARBUZOV, B., Prof.

"Why Minister's Order is Not Carried Out," by Professor B. Arbuzov, Corresponding Member of the USSR. Acad. of Sciences and Stalin Prize Winner.

SO: Izvestia, Sept 29, p. 2, 1951

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CA

Synthesis of alkoxydichlorostibines and dialkoxychlorostibines. B. A. Arbuzov and O. D. Samoilova (Kazan State Univ.). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1951, 717-21.—Heating 15.5 g. $(EtO)_3Sb$ with 15 g. PrOH to 90-115° gave 8 g. EtOH and 15.1 g. $(PrO)_3Sb$, b_{11} 115-16°, d_4^{20} 1.3841, n_D^{20} 1.4868. Addn. with cooling of 2.2 g. AcCl to 6.4 g. $(MeO)_3Sb$ gave a cryst. mass which on distn. gave 1.2 g. MeOAc and an unstated yield of $(MeO)_2SbCl$, b. 155-8°, m. 86-91°. Likewise, 8 g. $(MeO)_3Sb$ and 5.8 g. AcCl gave 7.4 g. $MeOSbCl_2$, b_{10} 120-1°, m. 36-9°, along with MeOAc. Similarly 10.4 g. $(EtO)_3Sb$ and 3.17 g. AcCl gave EtOAc and 5.2 g. (50.48%) $(EtO)_2SbCl$, b_{10} 119-20°, m. 35-8°; 8.8 g. ester and 5.38 g. AcCl similarly gave 66.2% $EtOSbCl_2$, b_{10} 114-18°, m. 69-71°. $(PrO)_3Sb$ (11 g.) and 2.9 g. AcCl gave PrOAc and 8 g. (86.4%) $(PrO)_2SbCl$, $b_{0.1}$ 123-9°, m. 50-1°; 8 g. ester and 4.2 g. AcCl gave 63.6% $PrOSbCl_2$, b_{10} 121-2.5°, m. 103-5°. $(BuO)_3Sb$ (6.5 g.) and 1.46 g. AcCl gave BuOAc and 48.8% $(BuO)_2SbCl$, b_{10} 150-1°, n_D^{20} 1.5312, which readily hydrolyzes in air; 13.5 g. ester and 6.4 g. AcCl gave 5.2 g. $BuOSbCl_2$, b_{10} 126-8°, m. 83-5°. $(C_6H_{13}O)_3Sb$ (10 g.) and 1.85 g. AcCl gave hexyl acetate, 2.1 g. $(C_6H_{13}O)_2SbCl_2$, b_5 151-3°, m. 53-5°, and 4.5 g. $(C_6H_{13}O)_2SbCl$, b_5 174-5°, n_D^{20} 1.5161. The former was obtained in 3.7-g. yield from 8 g. ester and 3 g. AcCl; the product b_5 144-6°, m. 59-5°. G. M. K.

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The parachor and the structure of esters of phosphoric, phosphorous, and thiophosphoric acid. B. A. Arbuzov and V. S. Vinogradova (Kazan State Univ.). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1951, 733–40; cf. *C.A.* 42, 3312e.—On the basis of parachor calcns. with corrections for chain parallelism (cf. Gibling, *C.A.* 36, 2355[5]), it is concluded that the $(RO)_3PO$ structure involves a parallel position of all 3 R chains. The thiophosphates, $(RO)_3PS$, possess a similar structure. However, phosphites, $(RO)_3P$, probably carry only 2 chains in parallel positions, indicating the necessity for a strongly polar group (PO or PS) to extend the structure of the entire mol. $POCl_3$ (10 g.), 20 g. $C_6H_{11}OH$, and 15.4 g. pyridine in 100 ml. Et_2O, with cooling, gave 7 g. $(C_6H_{11}O)_3PO$, b_7 187–8°, n_D^{20} 1.4340, d_4^{20} 0.9366, γ^{20} 28.04 (dynes/cm.), [*P*] 857.16 (within 0.1% of the calcd. value for 3 parallel chains). Similarly, 25 g. $C_6H_{11}OH$, 10 g. $POCl_3$, and 15.2 g. pyridine gave $(C_6H_{11}O)_2PO$, b_2 225–7°, n_D^{20} 1.4410, d_4^{20} 0.9200, γ^{20} 29.02, [*P*] 1094.7; prepn. with $PhNMe_2$ as the HCl-binding agent gave an undistillable product. Addn. of S to $(PrO)_3P$ gave $(PrO)_3PS$, b_{20} 123.5–4.5°, d_4^{20} 1.0177, n_D^{20} 1.4502, γ^{20} 28.47, [*P*] 545.4; heating $(BuO)_3P$ (20 g.) with 2.2 g. S to 180–200° gave $(BuO)_3PS$, b_{11} 158–9°, $b_{10.5}$ 160–1.5°, d_4^{20} 0.9871, n_D^{20} 1.4515, γ^{20} 26.36, [*P*] 660.2. Similarly were prepd. $(C_6H_{11}O)_3PS$, $b_{0.5}$ 188–8.5°, d_4^{20} 0.9501, n_D^{20} 1.4508, γ^{20} 28.68, [*P*] 892.6, and $(C_6H_{11}O)_2PS$, $b_{0.05\text{ to }0.1}$ 224–6° (distn. at higher pressure gives decompn.), d_4^{20} 0.9293, n_D^{20} 1.4593, γ^{20} 29.11, [*P*] 1126.4. BuOH, PCl_3, and pyridine gave $(BuO)_3P$, b_4 121.5°, d_4^{20} 0.9133, n_D^{20} 1.4327, γ^{20} 27.67, [*P*] 619.6. Similar reaction with $C_6H_{11}OH$ gave a mixt. of products which after repeated distn. yielded $(C_6H_{11}O)_3P$, b_2 167–8°, d_4^{20} 0.9002, n_D^{20} 1.4405, γ^{20} 27.86, [*P*] 853.5 (intermediate between 2 and 3 parallel chain structure); prepn. of the ester by heating 19.5 ml. $C_6H_{11}OH$ with 7.5 g. $(EtO)_3P$ and 1 drop H_3PO_4 (d. 1.8), with distn. of the EtOH, gave 5 g. pure $(C_6H_{11}O)_3P$, b_2 157–7.5°, d_4^{20} 0.8981, n_D^{20} 1.4428, γ^{20} 27.82, [*P*] 856.1 (very close to the structure requirement for 2 parallel chains). Similarly, $(C_6H_{11}O)_3P$, prepd. from PCl_3 (as above), b_1 210°, d_4^{20} 0.8936, n_D^{20} 1.4480, gave [*P*] 1084.1 (intermediate value), while that prepd. from $(EtO)_3P$, b_1 212–14°, d_4^{20} 0.8907, n_D^{20} 1.4475, γ^{20} 28.90, gave [*P*] 1089.8 (very close to 2-chain parallelism). Similarly $(C_6H_{11}O)_3P$ (from $(EtO)_3P$), b_1 226.5–7.5°, d_4^{20} 0.8879, n_D^{20} 1.4495, γ^{20} 29.39, gave [*P*] 1208 (very close to the requirement for 3-chain parallelism). $(C_6H_{11}O)_3P$, prepd. from PCl_3, $b_{7.1}$ 263°, d_4^{20} 0.8856, n_D^{20} 1.4500, γ^{20} 29.76, gave [*P*] 1325.7, while the product from $(EtO)_3P$, $b_{0.5}$ 236.5–7.5°, d_4^{20} 0.8842, n_D^{20} 1.4518, γ^{20} 29.99, gave [*P*] 1330.2 (very close to 2-chain parallelism). $PhCH_2CH_2OH$ (36 g.), 13 g. PCl_3, and 36 g. $PhNMe_2$ gave 18 g. $(PhCH_2CH_2O)_3P$, b_1 256–8°, d_4^{20} 1.1089, n_D^{20} 1.5595, γ^{20} 41.27, [*P*] not calcd. G. M. K.

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Dipole moments of esters of thiophosphoric acid. II. A. Arbuzov and T. G. Shavsha (Kazan State Univ.). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1951, 705-8; *Uchenye Zapiski Kazan. Gosudarst. Univ., Khimiya*, 110, No. 9, 99(1950); cf. *C.A.* 44, 8713g.—$(RO)_3PS$, prepd. by addn. of S to $(RO)_3P$, were examd. in C_6H_6 solns. The av. value of dipole moment was 3.04 D., close to that found in $(RO)_3PO$. Assuming OPO angle of 100° and OPS angle of 118°, taking OR dipole at 1.12 D. and PO dipole at 1.2 D., the value of the dipole moment of the PS link is 3.55 D., which is almost identical with that of PO (3.5 D.). $(EtO)_3PS$, b_{11} 94-5°, n_D^{20} 1.4488, d_4^{20} 1.0756, μ 2.91 D.; $(PrO)_3PS$, b_{10} 123.5-4.5°, n_D^{20} 1.4502, d_4^{20} 1.0177, μ 2.99 D.; $(BuO)_3PS$, b_{11} 158-9°, n_D^{20} 1.4515, d_4^{20} 0.9871, μ 3.04 D.; $(C_5H_{11}O)_3PS$, $b_{0.4}$ 188-8.5°, n_D^{20} 1.4552, d_4^{20} 0.9483, μ 3.18 D.; $(C_6H_{13}O)_3PS$, $b_{0.05-0.5}$ 224-6°, n_D^{20} 1.4592, d_4^{20} 0.9293, μ 3.08 D.

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ARBUZOV, B. A.

PA 195T12

USSR/Chemistry - Organic Antimony Compounds Sep/Oct 51

"Preparation of Antimonous Acid Esters by Re-esterification Reaction," B. A. Arbuzov, V. S. Vinogradova, Chem Sci Res Inst imeni A. M. Butlerov, Kazan' U imeni V. I. Ul'yanov-Lenin

"Iz Ak Nauk SSSR, Otdel Khim Nauk" No 5, pp 551-553

Found it possible to prep higher esters of antimonous acid by re-esterification of lower esters of antimonous acid with higher alcs and distng off of lower alcs. By this method prepd n-hexyl, n-octyl,

195T12

USSR/Chemistry - Organic Antimony Compounds (Contd) Sep/Oct 51

n-nonyl, n-decyl, benzyl, β-phenylethyl, and cyclohexyl esters of antimonous acid (none of these described in the literature).

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Action of triethyl phosphite and diethyl sodium phosphite on some dihalo derivatives. B. A. Arbuzov and B. P. Tugovkin (Kazan State Univ.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 99-107(1951).—To $(EtO)_3PONa$ (from 44.2 g. ester and 7.2 g. Na) in 450 ml. dry Et_2O was added 28.5 g. $ClCH_2CH_2CN$, b_{10} 63-5°, n_D^{20} 1.4370, d_4^{20} 1.1461, over 0.5 hr.; after refluxing 1 hr., the filtrate yielded 42.7 g. (70.2%) $(EtO)_2P(O)CH_2CH_2CN$, b_1 150-2°, b_4 127-8°, n_D^{20} 1.4380, d_4^{20} 1.1127; this (9.9 g.) heated 8 hrs. to 115-30° in a sealed tube with 30 ml. 2:1 HCl and evapd. gave 6.2 g. (78.5%) $(HO)_2P(O)CH_2CH_2CO_2H$, m. 166° (by Soxhlet extn. with dry C_6H_6, then with dry Me_2CO). Addn. of 40.6 g. $BrCH_2CHBrCN$, b_{10} 89-90°, n_D^{20} 1.5300, d_4^{20} 2.0955, to $(EtO)_3PONa$ (from 53.3 g. ester and 8.7 g. Na) in 500 ml. Et_2O and treatment as above gave after repeated fractionation 7 g. (11.9%) $(EtO)_2POH$, b_7 60-5°, n_D^{20} 1.4080, d_4^{20} 1.0807, and 9.5 g. (20.4%) $(EtO)_2P(O)C(CN):CH_2$ or $(EtO)_2P(O)CH:CHCN$, b_1 124-7°, n_D^{20} 1.4300, d_4^{20} 1.1277, sol. in H_2O and reacting rapidly with Br water or $KMnO_4$; hydrolysis with 1:1 HCl 10 hrs. at 150-65° gave on evapn. a mixt. of products, which, on removal of NH_4Cl by addn. of dry EtOH and evapn. of the ext., gave 4 g. oil; this with $BaCO_3$ gave a mixt. of Ba salts, from which was isolated a H_2O-insol. Ba salt, possibly $C_4H_8P_2O_6Ba_3$, $BaHPO_4$, or Ba phosphonoacetate; a considerable amt. of H_2O-sol. $C_4H_8O_6P_2Ba$ was also isolated. The products were not identified further. Addn. of 32.8 g. $(EtO)_3P$ to 20.2 g. $BrCH_2CHBrCN$ over 0.5 hr. led to a temp. rise to 87°, where it was kept for removal of the EtBr; after 15 min. at 150°, the mixt. yielded a total of 19 g. EtBr, but distn. of the residue failed to yield definite products. Addn. of 16.1 g. $PhCH:CHBr$, b_{10} 88-90°, n_D^{20} 1.6018, d_4^{20} 1.4094, to $(EtO)_2PONa$ (from 13.1 g. ester and 2 g. Na) in 100 ml. MePh (in Et_2O the reaction does not occur) gave no immediate NaBr, but after a 4-hr. reflux 0.6 g. NaBr pptd. and fractionation of the soln. gave 7.1 g. (44%) original bromide and 1.9 g. (12%) $PhCH(PO_3Et_2)CH_2P(O)(OEt)_2$, b_1 181°, n_D^{20} 1.4920, d_4^{20} 1.1524, which on hydrolysis 7 hrs. at 120-30° with 1:1 HCl gave the *free diphosphonic acid*, m. 212-14° (from H_2O). Possibly, both phosphono groups are attached to the terminal C atom, but no further characterization was attempted.

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PhCH:CHBr (14.9 g.) added to 14.5 g. $(EtO)_3P$ at 200–10° did not react and practically all the $(EtO)_3P$ was recovered; heating the components 8 hrs. to 180–200° in a sealed tube gave only traces of high-boiling products. Addn. of 34.6 g. $PhCHBrCH_2Br$ in Et_2O over 35 min. to $(EtO)_3PONa$ (from 37.5 g. ester and 6 g. Na) in 500 ml. Et_2O, gave a ppt. of NaBr and, after refluxing 2 hrs., 6.3 g. (53.7%) styrene, about 12 g. $(EtO)_3POH$, 4.8 g. unreacted dibromide, and about 14 g. of a fraction $b_{0.90}$ 132°, which on redistn. gave a liquid, b_1 105–130°; this with $2\text{-}C_6H_7NH_2$ gave 1.2 g. $(EtO)_2P(O)NHC_6H_7\text{-}2$, m. 69°, which indicated that the high-boiling liquid was probably $(EtO)_2POP(O)(OEt)_2$ (cf. Arbuzov and Arbuzov, C.A. 25, 3318). Addn. of 22 g. $(EtO)_3P$ over 1 hr. to 16 g. $PhCHBrCH_2Br$ preheated to 155–60° and heating 1 hr. to 165–70° gave 8.0 g. EtBr, 4.7 g. (50.2%) styrene, 3.8 g. crude $(EtO)_3POH$, b_1 69–72°, 1.7 g. unreacted dibromide, and 10.3 g. crude liquid, b_1 103–57°, which on redistn. gave a product, b_1 117–31°, forming with $2\text{-}C_6H_7NH_2$, 0.8 g. $(EtO)_2P(O)NHC_6H_7$, corresponding to a 4.8% yield of tetra-Et subphosphate. When $PhCHBrCH_2Br$ is heated alone to 155–70°, much HBr forms, thus explaining the formation of $(EtO)_3POH$ above. Heating 9.2 g. dibromide to 195–205°, gradual addn. of 10.2 g. $(EtO)_3POH$, and heating 1 hr. to 210–15° gave 4.1 g. EtBr and about 1.5 g. styrene (crude). Addn. of 21.4 g. PhCMe:CHBr, b_{11} 103–5°, n_D^{25} 1.5880, d_{25}^{25} 1.3790, to $(EtO)_3PONa$ (from 16 g. ester and 2.5 g. Na) in 200 ml. Et_2O and refluxing 1 hr. gave 2.1 g. unreacted bromide and 4 g. (16%) *PhCMe:CHP(O)(OEt)₂*, b_1 146–9°, n_D^{25} 1.5210, d_{25}^{25} 1.1094, which immediately reacts with Br; hydrolysis by 1:1 HCl 6 hrs. at 135–45° in a sealed tube and evapn. gave some crude PhCMe:CH_2 and H_3PO_4, isolated as the $PhNHNH_2$ salt, m. 155° (from EtOH). Addn. over 40 min. of 12.5 g. $(EtO)_3P$ to 13.6 g. PhCMe:CHBr preheated to 150–60° and heating 1 hr. to 165–70° gave 2.3 g. EtBr, 2.1 g. unreacted bromide, 3.1 g. $(EtO)_3POH$, and 5 g. (33.5%) *PhCMe:CHP(O)(OEt)₂*, $b_{0.5}$ 149–50°, n_D^{25} 1.5190, d_{25}^{25} 1.1000, which on hydrolysis with 1:1 HCl at 120–30° cleaves into H_3PO_4 and PhCMe:CH_2. $PhCMeBrCH_2Br$ (30.6 g.) with $(EtO)_3PONa$ (from 33 g. ester and 5.3 g. Na) in 400 ml. Et_2O, refluxed 2 hrs., gave 5.9 g. (45.4%) PhCMe:CH_2 and apparently about 6 g. $(EtO)_3POH$; no individual substances of higher b.p. were identified. $(EtO)_3PONa$ with $(MeCBr)_2$ yields apparently tetra-Et subphosphate and MeC:CMe.

G. M. Kosolapoff

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Addition of dialkyl phosphites to unsaturated compounds. I. Addition of dialkyl phosphites to 2,2-dimethylvinyl vinyl ketone. A. N. Pudovik and B. A. Arbuzov (State Univ., Kazan). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 382-8(1951); cf. *C.A.* 45, 2853*b*.—Treatment of 11 g. $Me_2C:CHCOCH:CH_2$ very slowly with a soln. of a very small piece of Na in 14 g. $(EtO)_2POH$ (much heat evolution, moderated by cooling), followed by 3 hrs. on a steam bath gave 13 g. crude product, b_5 150-65°, and 10 g. yellow viscous residue, which could not be distd. and which contained 13% P. Redistn. of the former product gave 52% *$Me_2C:CHCOCH_2CH_2PO(OEt)_2$*, b_5 149°, d_4^{20} 1.0858, n_D^{20} 1.4660; if the reaction is run in Et_2O the results are similar. Addn of 10 drops satd. EtOH-EtONa to 14 g. $(EtO)_2POH$ and 11 g. $Me_2C:CHCOCH:CH_2$ caused a violent reaction (further addn. does not cause heat evolution) and after 2 hrs. on a steam bath there was isolated 14 g. of the *above ester*, b_{11} 170-80°, n_D^{20} 1.4668, and 8.7 g. undistillable residue. The ester is a slightly yellow viscous fluid, sol. in H_2O, which readily adds Br, while hydrolysis with 1:1 HCl for 10 hrs. at 170-80° gave EtCl (3.3 g.) and on evapn. yielded the *free phosphonic acid*, an oil [since no H_3PO_4 was found, the α-phosphono position in respect to CO group is eliminated (*C.A.* 45, 7002*e*), and the above structure is confirmed], while addn. of *$PhNHNH_2$* to the acid gave a cryst. *addn. product*, m. 143-4° (from EtOH); oxidation with $KMnO_4$ in H_2O at room temp. gave Me_2CO and unidentified org. acids. Reaction of the *ester* with $PhNHNH_2$ gave the *phenylhydrazone*, $C_{18}H_{29}O_3N_2P$, b_2 210°. Addn. of 10 drops EtOH-EtONa to 11 g. $Me_2C:CHCOCH:CH_2$ and 11 g. $(MeO)_2POH$ produced a vigorous reaction and after 3 hrs. on a steam bath gave 13 g. *$Me_2C:CHCOCH_2CH_2PO(OMe)_2$*, b_{11} 169-71°, d_4^{20} 1.1130, n_D^{20} 1.4711. Similarly 14 g. $(iso\text{-}BuO)_2POH$ gave 10.7 g. *di-iso-Bu ester*, b_{11} 188-90°, d_4^{20} 1.0013, n_D^{20} 1.4511, while 10 g. ketone and 18 g. *$(BuO)_2POH$* gave 16 g. *di-Bu ester*, b_{11} 198-200°, n_D^{20} 1.4623, d_4^{20} 1.0111. Much more EtONa is needed to complete the reactions of the higher esters. G. M. Kosolapoff

1951

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

Addition of dialkylphosphorous acids (dialkyl phosphites) to unsaturated compounds. II. Addition of dialkylphosphorous acids to acrylonitrile and to methyl methacrylate. A. N. Pudovik and B. A. Arbuzov (A. E. Arbuzov Chem. Inst., Kazan). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 1837–41(1951); cf. *C.A.* 46, 1962a.—To 10.8 g. CH_2:CHCN (I) and 22 g. $(MeO)_2POH$ was slowly added dropwise with cooling a satd. soln. of MeONa in alc. (presumably MeOH); the 1st few drops caused a very vigorous reaction (even with cooling, a temp. of 60–70°); several drops (amt. unstated) are necessary for the completion of reaction, and further addn. does not cause heat evolution. Distn. yielded 28.3 g. $(MeO)_2P(O)CH_2CH_2CN$, bₚ 158°, n_D^{20} 1.4432, d_4^{20} 1.1964. This (8 g.) heated 10 hrs. with 30 ml. 1:1 HCl in a sealed tube gave, on evapn. to dryness, re-evapn. with H_2O, and extn. of the residue with Me_2CO, $HO_2CCH_2CH_2PO(OH)_2$, m. 164–5° (from Me_2CO). Addn. of 10 drops satd. MeONa in MeOH to 7 g. I and 12 g. $(EtO)_2POH$ with cooling similarly gave 15.8 g. (83%) $(EtO)_2P(O)CH_2CH_2CN$, bₚ 126–9°, n_D^{20} 1.4388, d_4^{20} 1.1080. Likewise 5 g. I and 17 g. $(iso\text{-}BuO)_2POH$ with a much larger amt. of MeONa-MeOH (actual amt. unstated) gave 15.6 g. (71%) $(iso\text{-}BuO)_2P(O)CH_2CH_2CN$, bₚ 171°, n_D^{20} 1.4396, d_4^{20} 1.0830. Similarly, 7 g. I, 29 g. $(BuO)_2POH$, and 2 ml. satd. MeONa-MeOH gave 23.6 g. (71.5%) $(BuO)_2P(O)CH_2CH_2CN$, bₚ 177–8°, n_D^{20} 1.4365, d_4^{20} 1.0655. Also prepd. in 46.4% yield was $(iso\text{-}PrO)_2P(O)CH_2CH_2CN$, bₚ 160°, n_D^{20} 1.4315, d_4^{20} 1.0900. The reaction of 9 g. $(MeO)_2POH$ with 9 g. MeO_2CCMe:CH_2 (II) required the addn. of 1–1.5 ml. satd. MeONa-MeOH before action commenced and it proceeded with much vigor; distn. yielded 14 g. (77.7%) $(MeO)_2P(O)CH_2CHMeCO_2Me$, bₚ 137–8°, n_D^{20} 1.4377, d_4^{20} 1.1761. II (6.5 g.) and 8 g. $(EtO)_2POH$ similarly gave 10.8 g. (74.5%) $(EtO)_2P(O)CH_2CHMeCO_2Me$, bₚ 152–3°, n_D^{20} 1.4350, d_4^{20} 1.1212. Similarly was obtained 51% $(iso\text{-}PrO)_2P(O)CH_2CHMeCO_2Me$, bₚ 153–4°, n_D^{20} 1.4298, d_4^{20} 1.0612. The reaction is believed to proceed by an ionic route with addn. of $(RO)_2PO^-$ to the pos. end of the unsatd. link and Na going to the neg. pole, and a final mole of $(RO)_2POH$ replacing the Na with H. G. M. K.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

Synthesis of esters of phosphonic acids containing heterocyclic radicals. I. Synthesis of esters of phosphonic acids with a thiazole radical. B. A. Arbuzov and B. P. Lugovkin (Kazan State Univ.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 1869-72 (1951).—To 9.7 g. $(EtO)_3P$ preheated to 150-5° was added dropwise 8.2 g. 4-chloromethyl-2-methylthiazole, b_5 73-5°, n_D^{20} 1.5440 (cf. Suter and Johnson, *C.A.* 25, 952; U.S. patent 2,014,498, *C.A.* 29, 7344[8]) over a period of 0.5 hr.; after 1 hr. at 160-70° distn. of the soln. gave 9.5 g. (68.1%) *di-Et 2-methyl-4-thiazolylmethanephosphonate*, b_5 147-50°, d_{20}^{20} 1.1892, n_D^{20} 1.5030. Heating the ester in a sealed tube with 1:1 HCl 6 hrs. at 110-25°, clarification with charcoal, and repeated evapn. with H_2O gave 2.1 g. crude cryst. *HCl salt* of the corresponding *phosphonic acid*, which on crystn. from hot H_2O and drying at 110-15°, m. 142°, resolidified at 162°, and remelted at 167°; the product contained 4% less Cl than theoretical. Similarly 5.2 g. $(EtO)_3P$ with 4.9 g. 4-chloromethyl-2-ethylthiazole at 170-80° gave 5.4 g. (67.5%) *di-Et 2-ethyl-4-thiazolylmethanephosphonate*, b_5 148-50°, d_{20}^{20} 1.1655, n_D^{20} 1.5000; hydrolysis as above gave again the crude *free acid HCl salt*, m. 130-5° after drying at 110-30°. 4-Chloromethyl-2-phenylthiazole and $(EtO)_3P$ gave, after 0.5 hr. at 140-50° and 1 hr. at 170-80°, 85.3% *di-Et 2-phenyl-4-thiazolylmethanephosphonate*, b_5 210-11°, d_{20}^{20} 1.2153, n_D^{20} 1.5650; *free acid HCl salt* m. 180-1° (from H_2O). 4-Chloromethyl-2-(*p*-methoxyphenyl)thiazole gave 73.7% *di-Et 2-(p-methoxyphenyl)-4-thiazolylmethanephosphonate*, b_5 230-2°, m. 47-8° (from Et_2O); *free acid HCl salt*, m. 181-6° (from H_2O). While 4-chloromethyl-2-aminothiazole-HCl reacted with $(EtO)_3P$, no pure products could be isolated.

G. M. Kosolapoff

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CA

Action of acrylonitrile and acrolein on α- and β-pyronenes. B. A. Arbuzov and A. R. Vil'chinskaya (Kazan State Univ.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 1872-8(1951).—Alloöcimene passed over glass chips at 400-20° yielded among minor by-products (p-cymene, etc.) *α-pyronene*, b_{10} 30-1°, d_4^{20} 0.8284, n_D^{20} 1.4600, and the *β-isomer*, b_{10} 35-6°, d_4^{20} 0.8493, n_D^{20} 1.4795. Heating the former (31.4 g.) with 14.4 g. acrolein 12 hrs. to 100° gave about 18 g. $C_{10}H_{16}O$, b_4 108-9°, d_4^{20} 0.9764, n_D^{20} 1.4895; this *aldehyde* forms a *semicarbazone*, m. 226°. *β-Pyronene* in the presence of hydroquinone similarly gave an *aldehyde* in the form of a waxy mass, b_4 104-5°, b_1 110-11°, b_{10} 120°, forming a *semicarbazone*, m. 209-10° (cf. Dupont and Dulou, *C.A.* 30, 7575[5]; 33, 9312[2]). The 1st aldehyde (23.8 g.) added to 153 g. $AcCH_2CO_2Et$ treated with 6 g. Na, stirred 2.5 hrs., and acidified with AcOH gave a product $C_{18}H_{26}O_3$, b_4 154-7°, d_4^{20} 0.9864, n_D^{20} 1.4935. Heating equal wts. of α-pyronene with CH_2:CHCN and a little hydroquinone 20 hrs. at 125-40° gave about 30% adduct, $C_{13}H_{19}N$, b_{10} 128-30°, d_4^{20} 0.9574, n_D^{20} 1.4875. The *β-isomer* similarly gave an *adduct*, wax, m. 45-50°, b_4 125-7°. Reduction of the 1st adduct with Na-EtOH gave an amine, $C_{13}H_{23}N$, b_{10} 138-40°, d_4^{20} 0.9450, n_D^{20} 1.4995 (*picrate*, m. 187-8°), while the 2nd product gave an *amine*, $C_{13}H_{23}N$, b_{10} 125-6°, d_4^{20} 0.9470, n_D^{20} 1.5010 (*picrate*, m. 199-201°). No decisions as to structures of the products are made. G. M. Kosolapoff

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ARBUZOV, B. A.

CA

Dipole moments and structure of esters of orthocarbonic, orthosilicic, and orthotitanic acids. B. A. Arbuzov and T. G. Shavsha (Kazan State Univ.). *Doklady Akad. Nauk S.S.S.R.* 79, 599-600(1951); cf. *C.A.* 44, 886*e*.—The previously detd. exptl. dipole moments μ of CO_4R_4 (I) (μ = 1.1 D.), SiO_4R_4 (II) (1.66), and TiO_4R_4 (III) (1.65) are compared with values calcd. on the assumption of definite structures. For a tetrahedral model with completely free rotation of the OR groups, the calcd. μ = 1.70 or 1.78, depending on the formula used (Zahn or Lander). Four space models are possible with hindered rotation. Of these, 2 give μ = 3.18 and 3.83, resp., and consequently are out of consideration. In a 3rd model, 2 chains lie in the plane of the drawing and 2 in a perpendicular plane, giving μ (calcd.) = 1.62. A 4th structure, with 3 chains in a plane perpendicular to the plane of the drawing and 1 OR chain rotating freely, gives a calcd. μ = 1.1. With the at. radii C 0.77, O 0.66, Si 1.17, Ti 1.80 A., and a van der Waals radius of CH_3 = 2.0 A., free rotation of the OR group is evidently impossible in I and II owing to superposition of the van der Waals force fields; there is some superposition also for III. Comparison of the calcd. and the exptl. μ for I excludes the 1st 3 structures and leaves only the 4th structure with μ = 1.1. For II, the 3rd and the 4th structure are possible, but only the 3rd gives good numerical agreement in μ. For III, the 3rd rigid structure and free rotation are possible. For I and II, these conclusions are in accord or compatible with the parachor data of Arbuzov and Vinogradova (*C.A.* 42, 8559*d*) which are definitely in favor of pairwise disposition of the OR chains in II (3rd structure) although they permit no discrimination between the 3rd and the 4th structure in II. Absence of free rotation of OR in II is also indicated by electron diffraction in $Si(OMe)_4$ (Yamazaki, *et al.*, *C.A.* 44, 10437*d*).

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2

Development of organic chemistry in light of A. M Butlerov's teachings. B. A. Arbuzov. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1952, 14-21. - Historical review.

G. M. Kosolapoff

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.; GRECHKIN, N.P.

Synthesis of benz[a]anthracene-7-methanephosphonic and 12-methylbenz[a]-anthracene-7-methanephosphonic acid. Bull. Acad. Sci. U.S.S.R., Div. Chem. Sci. '52, 131-3 [Engl.translation].
(CA 47 no.19:9931 '53)

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

Chemical Abst.
Vol. 48 No. 9
May 10, 1954
Organic Chemistry

2

2 Chem

Cetyl esters of some inorganic acids. B. A. Arbuzov N. S. Vinogradova. *Bull. Acad. Sci. U.S.S.R., Div. Chem. Sci.* 1952, 483-4(Engl. translation).—See *C.A.* 47, 4836d.
H. L. H.

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3

Chemical Abst.
Vol. 48 No. 9
May 10, 1954
Organic Chemistry

② Chem

Viscosity and structure of dialkyl phosphites. B. A. Arbuzov and V. S. Vinogradova. *Bull. Acad. Sci. U.S.S.R., Div. Chem. Sci.* 1952, 485-9(Engl. translation). See *C.A.* 47, 4834d. H. L. H.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

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

(2) Chem

Diethylamides of esters of phosphonoformic acid. B. A. Arbuzov and N. T. Rizpolozhenskii. *Bull. acad. sci. U.S.S.R., Classe sci. chim.* 1952, 759-64 (Engl. translation). See *C.A.* 47, 10457g. H. L. H.

11-11-54

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

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

Esters of ethylphosphonous acid and some of their transformations. I. Complete esters of ethylphosphonous acid. B. A. Arbuzov and N. I. Riznopolskii. *Bull. acad. sci. U.S.S.R., Classe sci. chim.* 1952, 765-72 (Engl. translation). See *C.A.* 47, 9903c. II. Incomplete esters of ethyl phosphonous acid. *Ibid.* 843-6. See *C.A.* 47, 9904c. H. L. H.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

(2)

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

Viscosity and structure of esters of phosphorous, phosphoric, and thiophosphoric acids and boric acid. B. A. Arbuzov and V. S. Vinogradova. *Bull. acad. sci. U.S.S.R., Classe sci. chim.* 1952, 773-80(Engl.translation). See *C.A.* 47, 10458e. H. L. H.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

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

(2) 5 1

Dipole moments of diesters of diethylamidophosphoroformic acid. B. A. Arbuzov and T. G. Shavsha. *Bull. acad. sci. U.S.S.R., Classe sci. chim.* 1952, 781-5(Engl. translation). See *C.A.* 47, 10458*c*. H. L. H.

MF 4-28-54

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

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

(2) 4

Esters of alkylphosphonic acids and their parachors. B. A. Arbuzov and V. S. Vinogradova. *Bull. acad. sci. U.S.S.R., Classe sci. chim.* 1952, 787-95(Engl. translation). See *C.A.* 47, 10464h.
H. L. H.

AF 9-28-54

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.; RIZPOLOZHENSKIY, N.I.

Diethylamides of esters of phosphonoformic acid. Izvest. Akad. Nauk S.S.S.R. Otdel. Khim. Nauk '52, 847-53. (MLRA 5:11)
(CA 47 no.20:10457 '53)

1. A.E.Arbuzov Inst., Kazan.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.; RIZPOLOZHENSKIY, N.I.

Esters of ethylphosphonous acid and some of their transformations. I. Complete esters of ethylphosphonous acid. Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk '52, 854-64. (MIRA 5:11)
(CA 47 no.19:9903 '53)

1. A.Ye.Arbuzov Inst., Kazan.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.; VINOGRADOVA, V.S.

Viscosity and structure of esters of phosphorous, phosphoric, and thiophosphoric acids and boric acid. Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk '52, 865-74.
(CA 47 no.20:10458 '53)

1. Kazan State Univ.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.; SHAVSHA, T.G.

Dipole moments of diesters of diethylamidophosphonoformic acid. Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk '52, 875-81. (MLRA 5:11)
(CA 47 no.20:10458 '53)

1. Kazan State Univ.

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AKBUZOV, B.A.; VINOGRADOVA, V.S.

Esters of alkylphosphonic acids and their parachors. Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk '52, 882-93. (MLRA 5:11)
(CA 47 no.20:10464 '53)

1. Kazan State Univ.

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

Results of the All-Union conference on the theory of chemical structure in organic chemistry. Khim. v shkole, no. 1, '52.

SO: MLRA, October 1952

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

USSR/Chemistry - Cancerogenic Substances Jan/Feb 52

"Synthesis of 1,2-Benzanthryl-10-Methylphosphonic and 1,2-Benz-9-Methylanthryl-10-Methylphosphonic Acid," B. A. Arbuzov, Chem Inst imeni A. Ye. Arbuzov, Kazan Affiliate, Acad Sci USSR

"Iz Akad Nauk, Otdel Khim Nauk" No 1, pp 121-123

Phosphonic acids in question and their ethyl esters were prepd by treating 10-chloromethyl-1,2-benzanthracene, and 9-methyl-10-iodomethyl-1,2-benzanthracene resp with diethylphosphorous acid sodium salt. Compds obtained are derivs of active cancerogenous compds.

208T9

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV , B. A.

USSR/Chemistry - Cetyl Esters May/Jun 52

"Cetyl Esters of Some Inorganic Acids,"
B. A. Arbuzov, V. S. Vinogradova, Sci
Res Chem Inst imeni A. M. Butlerov,
Kazan' State U imeni V. I. Ulyanov-Lenin

"Iz Ak Nauk, Otdel Khim Nauk" No 3, pp 505,506

By re-esterification with cetyl alc of ethyl esters the following products were obtained: tricetylphosphite and tetracetylorthosilicate. Tricetyl borate was obtained by the action of cetyl alc on boric acid. All esters obtained can be distilled in vacuum without decompg.

220T16

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

USSR/Chemistry - Organo-Phosphorus Compounds May/Jun 52

"The Viscosity and Structure of Dialkylphosphorous Acids," B. A. Arbuzov, V. S. Vinogradova, Sci Res Chem Inst imeni A. M. Butlerov, Kazan State U imeni V. I. Ulyanov-Lenin

"Iz Ak Nauk, Otdel Khim Nauk" No 3, pp 507-511

Detn of the viscosity of dialkylphosphorous acids in dild solns of carbon tetrachloride or benzene showed that according to the given values of η equiv they are in the monomeric state. The following compds were synthesized for the 1st time by re-esterification of diethylphosphorous acid with the corresponding alc: dinonylphosphorous, didecylphosphorous, decetylphosphorous, di-β-phenylethylphosphorous, ethylhexylphosphorous and ethyloctylphosphorous acids.

220T17

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

1. ARBUZOV, B. A.; VINOGRADOVA, V. S.

2. USSR (600)

4. Esters

7. Viscosity and structure of esters of phosphorous, phosphoric, thiophosphoric and boric acids, Izv. AN SSSR. Otd. khim. nauk, No. 5, 1952.

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

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

1. ARBUZOV, B. A., RIZPOLOZHENSKIY, N. I.

2. USSR (600)

4. Phosphinic Acid

7. Esters of ethylphosphinic acid and their transformations. Part 2. Incomplete esters of ethylphosphinic acid. Izv. AN SSSR. Otd. khim. nauk, No. 5, 1952

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

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.

USSR/Chemistry - Organotitanium Compounds Apr 52

"Preparation of Esters of Orthotitanic Acid by Re-Esterification," B. A. Arbuzov, Z. G. Isayeva, Sci Res Inst imeni A. M. Butlerov, Kazan State U

"Zhur Obshch Khim" Vol XXII, No 4, pp 566, 567

Re-esterification of the ethyl ester of orthotitanic acid produced a high yield of the n-butyl, n-hexyl, n-octyl, and n-nonyl esters of orthotitanic acid.

Chem. Abst. Vol. 48, No. 5 - Mar. 10, 1954

224T30

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

USSR /Organophosphorus Compounds Jul 52

"The Synthesis of Esters of Phosphonic Acids Containing Heterocyclic Radicals," II. Ethyl Esters of Phosphonic Acids With Oxygen-Containing Heterocyclic Radicals," B. A. Arbuzov, B. P. Lugovkin, Sci Res Chem Inst imeni A. M. Butlerov, Kazan' State U

"Zhur Obshch Khim" Vol 22, No 7, pp 1193-1198

By the action of triethylphosphite or sodium diethylphosphite on corresponding halogen derivs, representatives of esters of phosphonic acids were

229T42

prepd with heterocyclic radicals contg 1 or 2 oxygen atoms. Esters of phosphonic acids were synthesized which contain 3-,5-,or 6-membered rings with a single atom of oxygen, or a 5-membered dioxolane ring with 2 atoms of oxygen. Sapon of the prepd esters was carried out.

Synthesis of other heterocyclic compounds are also described.

229T42

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

USSR/Chemistry - Organophosphorus Compounds Jul 52

"The Synthesis of Esters of Phosphonic Acids, Containing Heterocyclic Radicals," III. The Action of Sodium Diethyl Phosphite and Triethyl Phosphite on Some Halogen Derivatives of Pryidine and Piperidine," B. A. Arbuzov, B. P. Lugovkin, Sci Res Chem Inst imeni A. M. Butlerov, Kazan' State U

"Zhur Obshch Khim" Vol 22, No 7, pp 1199-1204

By the action of sodium diethylphosphite on N-(β-bromethyl)-piperidine and N-(γ-brompropyl)-

229T43

piperidine, the ethyl esters of N-(β-phosphonicacid ethyl) piperidine and N-(γ-phosphonicacid propyl)-piperidine were synthesized. The chlorides of the corresponding acids were prepd by the sapon of these esters. The action of triethylphosphite on N-(β-bromoethyl)-piperidine and N-(γ-bromopropyl)-piperidine led to the formation of high-melting substances of obscure structure. α-Bromine and the α-iodopyridines did not react with sodium diethylphosphite. α-Vinylpyridine was apparently the principal product of the reaction of sodium diethylphosphite with 2-(β-bromoethyl)-pyridine.

229T43

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ARBUZOV, B.A.

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Chem

Chemical Abst.
Vol. 48 No. 5
Mar. 10, 1954
Organic Chemistry

Synthesis of esters of phosphonic acids containing heterocyclic radicals. II. Ethyl esters of phosphonic acids with oxygen-bearing heterocyclic radicals. B. A. Arbuzov and B. P. Lugovkin (Kazan State Univ.). *J. Gen. Chem. U.S.S.R.* 22, 1241-6(1952)(Engl. translation).—See *C.A.* 47, 4871*c*. III. The action of sodium diethyl phosphite and triethyl phosphite on some halogen derivatives of pyridine and piperidine. *Ibid.* 1247-51.—See *C.A.* 47, 4872*e*. H. L. H.

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APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.; FRINOVSKAYA, V.A.

Dichloride of d-Δ^3-carene. Zhur. Obshchey Khim. 22, 1444-45 '52. (CA 47 no.13:6379 '53) (MLRA 5:8)

1. Kazan. Sate Med. Inst.

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.

(2) 3

Chemical Abst.
Vol. 48 No. 5
Mar. 10, 1954
Organic Chemistry

Dichloride of d-Δ³-carene. B. A. Arbuzov and V. A. Prinovskaya. *J. Gen. Chem. U.S.S.R.* 22, 1487-8(1952) (Engl. translation).—See *C.A.* 47, 6379f. H. L. H.

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.; ISAYEVA, Z.G.

Some reactions of products of cyanoehtylation of dihydric phenols by acrylonitrile. Zhur. Obshchey Khim. 22, 1645-7 '52. (MLRA 5:9)
(CA 47 no.17:8681 '53)

1. V.I. Ul'yanov-Lenin State Univ., Kazan.

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

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

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Some reactions of products of cyanoethylation of dihydric phenols by acrylonitrile. B. A. Arbuzov and L. G. Isaeva. *J. Gen. Chem.* (U.S.S.R.) 21, 1685-7 (1952) (Engl. translation).—See *C.A.* 47, 8681*i*. H. L. H.

9-2-54

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

USSR/Chemistry - Cancerogenic Compounds Sep 52

"Synthesis of 3,4-Benzopyrene and 3,4,6,7-Dibenzopyrene Derivatives Through Phenalone-9 and Benzanthrone," B. A. Arbuzov, N. P. Grechkin, Chem Inst imeni A. Ye. Arbuzov, Kazan' Affiliate, Acad Sci USSR

"Zhur Obshch Khim" Vol 22, No 9, pp 1692-1700

232T34

3,4,6,7-Dibenzopyrene and 1',2',3,4-naphtho-6,7-benzopyrene both possess strong blastomogenic action; this prompted the synthesis of derivs of 3,4,6,7-dibenzopyrene. By treating benzanthrone with organomagnesium compds contg methyl or methylene groups in the ortho position, the corresponding 4-substituted benzanthrones were prepared. Pyrolysis of the latter yielded derivs of 3,4,6,7-dibenzopyrene. In this manner, the following were prepd for the 1st time: 3-methyl-3,4,6,7-dibenzopyrene, 1',3'-dimethyl-3,4,6,7-dibenzopyrene, 2'isopropyl-3,4,6,7-dibenzopyrene, 3'-methyl-4',5-ethylene-3,4,6,7-dibenzopyrene, and 3,4,6,7-tribenzopyrene. In an analogous manner 3,4-benzopyrene and 1',2',3,4-naphthopyrene were prepd by starting with phenalone-9 through 1-substituted phenalones. The high cancerogenic activity of 3,4,6,7-dibenzopyrene and the lack of activity in 2',3'-naphtho-3,4-pyrene do not agree with the statement of A. Pullman concerning the connection between cancerogenic activity and the presence of a specific "K" field in the mol of the cancerogenic substance which possesses an increased density of Pi electrons.

(CA 47 no. 19: 9913 '53)

ARBUZOV, B. A.

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232T34

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

USSR/Chemistry - Organophosphorus Compounds Nov 52

238T38

"The Alkylation of Amines With Chlorides of Dialkylphosphoric Acid," B. P. Lugovkin and B. A. Arbuzov, Chem Inst imeni A. M. Butlerov, Kazan' State U imeni V. I. Lenin

"Zhur Obshch Khim" Vol 22, No 11, pp 2041-2047

The action of chlorides of diethylphosphoric acid and dibutylphosphoric acid on carbazole was studied. The heating of these components above 200° resulted in a reaction which produced N-ethyl- and N-butylcarbazoles, resp. The alkylating action of the chlorides of dialkylphosphoric acid was also detd on the examples of aniline, β-naphthylamine,

238T38

diphenylamine and α-aminopyridine. The reaction resulted in N-dietnylaniline(87%), N-diethyl-β-naphthylamine(88%), N-ethyldiphenylamine(83%), N-methyldiphenylamine(69%) and N-methylpyridonimine(29%), resp. Alkylation with acid chlorides of dialkylphosphoric acid was shown to take place also in the case of amides of acids. To explain the mechanism of the alkylating action of the acid chlorides, a study was made of the effect of heat on the anilide of diethylphosphoric acid. It was demonstrated that when the latter was heated up to 230°, diethylaniline (85%) was formed. A preliminary scheme for the fission of the anilide of diethylphosphoric acid is given.

238T38

(CA 47 no. 18:9283 '53)

ARBUZOV B. A.

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ARBUZOV, B. A.

Chemical Abst.
Vol. 48 No. 9
May 10, 1954
Organic Chemistry

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Chem.

Alkylation of amines by chlorides of dialkyl phosphates. B. P. Lugovkin and B. A. Arbuzov. *J. Gen. Chem. U.S.S.R.* 22, 2095-2101(1952)(Engl. translation).—See *C.A.* 47, 9283*h*.

H. L. H.

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.

USSR/Chemistry-Amines

"Parachor and Structure of Secondary Amines," B.A.Arbuzov and L. M. Kateheva; Chem Inst. im A.M.Butlerov, Kazan State 4

Zhur Fiz Khim, Vol 26, No.9, pp. 1298-1302, 1952

The parachors of seven secondary amines were detd and the discosity of del benzene solns of five secondary amines was measured. Both the parachor method and the viscometric method indicated that the most probable structure of the mols of secondary alophatic and aromatic amines is the structure with hydrocarbon chains pointing in opposite directions from the N atom

(CA 47 no. 22: 12219 '53)

263 T 20

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B. A.

234T2

USSR/Chemistry - Organophosphorus Compounds 1 Mar 52

"Preparation of Higher Dialkylphosphorous Acids by the Reaction of Re-esterification," B. A. Arbuzov, Corr Mem, Acad Sci USSR, V. S. Vinogradova, Sci Res Inst imeni A. M. Butlerov, Kazan State U imeni V. I. Ul'yanov-Lenin

"Dok Ak Nauk SSSR" Vol 83, No 1, pp 79, 80

Two moles of the appropriate alc were reacted with one mole of diethylphosphorous acid in the presence of a few drops of concd phosphoric

234T2

acid catalyst to form the following dialkylphosphorous acids, which were isolated: dihexyl, dioctyl, dionyl, didecyl, dicetyl, and di-beta-phenylethyl. Hexylethyl and octylethyl phosphorous acids were obtained by using only one mole of the alc. Re-esterification can also be carried out with the following alcs: ethylene glycol, cycolhexanol, benzylalc, and octanol-2. However, distn caused the products to decompose, so that isolation of esters of this group was unsuccessful.

234T2

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

PA 234T10

USSR/Chemistry - Organophosphorus Compounds 1 Apr 52

"Esters of Ethylphosphinic Acid," B. A. Arbuzov, N. I. Rizpolozhenckiy, Chem Inst imeni A. Ye. Arbuzov, Kazan' Affiliate, Acad Sci USSR

"Dok Ak Nauk SSSR" Vol 83, No 4, p 581

The following esters of ethylphosphinic acid have been synthesized for the 1st time: dimethyl, diethyl, dipropyl, diisopropyl, dibutyl, dihexyl, and dioctyl. They were obtained through the action of the appropriate alc on ethyldichlorphosphine in the presence of pyridine. The reaction was carried out in ether as a solvent under an atm of carbon dioxide. Tabulates the phys properties and yields of these compds which oxidize very readily in air. Investigation of the esters is being continued.

ARBUZOV, B. A.

234T10

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CA

General & Physical Chemistry 2

Dipole moments of some complex compounds of neutral esters of phosphorous acid. B. A. Arbuzov and T. G. Shavsha (Kazan State Univ.). *Doklady Akad. Nauk S.S.S.R.* 84, 507-8(1952).—Detns. of dipole moments in C_6H_6 soln. were made on the following complexes (dipole moment in D. of the entire substance and that of the phosphite ester alone, resp.): $CuCl.2P(OPh)_3$ 1.70 and 1.00; CuBr analog, 1.65 and 1.00; CuI analog, 100 and 1.00; $[CuCl.P(OPh)_3]_2$, 3.21 and 1.00; CuBr analog, 3.39 and 1.00; $[CuI.P(O\ iso\text{-}Pr)_3]_2$, 2.57 and 1.98; CuBr analog, 2.78 and 1.98; AgI analog, 3.43 and 1.98; AgBr analog, 3.53 and 1.98; $AuCl.P(OMe)_3$, 7.08 and 1.83; $AuCl.P(OPh)_3$, 6.27 and 1.00; $[PtCl_2.PCl_3]_2$, 3.08 and 1.10; $[PtCl_2.P(OMe)_3]_2$, 4.02 and 1.83; $PtCl_2.2P(OMe)_3$, 9.23 and 1.83; $PtCl_2.2P(O\ iso\text{-}Pr)_3$, 9.05 and 1.98; $PtCl_2.2P(OEt)_3$, 9.12.—. The results from the 1st 3 substances do not agree with the suggestion of Mann, *et al.* (*C.A.* 31, 1720[8]) concerning the bridged character of complexes $CuI.2PEt_3$. Compds. of type $CuX.P(OR)_3$ have a trimeric mol.; analogous complexes of R_3P are given a tetrahedral structure by Mann, *et al.* (*loc. cit.*) which does not agree with the high dipole moment of such complexes (1.6D. for $CuI.Bu_3As$). The complexes with AuCl have high moment and are apparently linear. The 3 last complexes appear to have cis configuration; their dipole moment is close to that of *cis*-chloride, $PtCl_2.2PCl_3$. This chloride complex and its $P(OMe)_3$ analog are probably of bridged type; Chatt (*C.A.* 45, 0952c) suggests trans structure for such compds. based on R_3P.

G. M. Kosolapoff

APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4"

"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

ARBUZOV, B.A.

MERLIN, V.S.; MARTYNOV, D.Ya., otvetstvennyy redaktor; MARKOV, M.V., professor, redaktor; SHAFUGULLIN, A.G., professor, redaktor; ARBUZOV, B.A., professor, redaktor; DIUKOV, I.A., professor, redaktor; NORDEN, A.G., professor, redaktor; PISAREV, V.I., professor, redaktor; TIKHVINSKAYA, Ye. I., professor, redaktor; ABDRAKHAMANOV, M.I., dotsent, redaktor; MOROZOV, D.G., dotsent, redaktor; KHARITONOV, A.P., dotsent, redaktor; KOLOBOV, N.V., redaktor; KOLESNIKOVA, Ye.A., starshiy prepodavatel', redaktor; ROZHDESTVENSKIY, B.P., dotsent, redaktor.

[Peculiarity of conditioned reactions in the structure of a voluntary act] Svoeobrazie uslovnykh reaktsii v strukture volevogo akta. Kazan', 1953. 123 p. (Kazan. Universitet. Uchenye zapiski, vol.113, no.3) (MLRA 10:3)

1. Rektor universiteta (for Martynov); 2. Prorektor po nauchnoy rabote (for Markov); 3. Prorektor po uchebnoy rabote (for Shafugullin). 4. Sekretar' partbyuro universiteta (for Kolobov)

(CONDITIONED RESPONSE) (WILL)

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"APPROVED FOR RELEASE: 06/05/2000 CIA-RDP86-00513R000101920004-4

MADANOV, P.V.; MARTYNOV, D.Ya., otvetstvennyy redaktor; MARKOV, M.V., professor, redaktor; SHAFUGULLIN, A.G., professor, redaktor; ARBUZOV, [redacted], akademik, redaktor; DYUKOV, I.A., professor, redaktor; NORDEN, A.P., professor, redaktor; PISAREV, V.I., professor, redaktor; TIKHVINSKAYA, Ye.I., professor, redaktor; ABDRAKHMANOV, M.I., dotsent, redaktor; MOROZOV, D.G., dotsent, redaktor; KHARITONOV, A.P., dotsent, redaktor; KOLOBOV, N.V., redaktor; KOLESNIKOVA, Ye.A., starshiy prepodavatel', redaktor; VINOKUROV, M.A., professor, redaktor.

[Biological accumulation of manganese in soils of the Volga-Kama forest-steppe and its availability to plants] Biologicheskaia akkumuliatsiia marganetsa v pochvakh Volzhsk-Kamskoi lesostepi i ego dostupnost' sel'skokhoziaistvennym rasteniiam. Kazan', 1953. 202 p. (Kazan. Universitet. Uchenye zapiski, vol.113, no.7) (MIRA 10:3)

1. Rektor universiteta (for Martynov). 2. Prorektor po nauchnoy rabote (for Markov). 3. Prorektor po uchebnoy rabote (for Shafugullin) 4. Sekretar' partbyuro universiteta (for Kolobov).
(Plants, Effect of manganese on)
(Volga Valley—Forest soils)

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ARBUZOV, B.A.

USSR.

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