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Mobility of the halogen in bromonitronaphthalenes. Yu. S. Zalkind. *Zhur. Obshchei Khim.*, Khim. Ser., 1, 151-62(1931); cf. *C. A.* 25, 3129.—The reactivity of the Br atom in bromonitronaphthalenes is the subject of the investigation, which was begun by refluxing 1,4-$C_{10}H_6BrNO_2$, 1,5-$C_{10}H_6BrNO_2$ and 1,8,4-$C_{10}H_5(NO_2)_2Br$ with EtONa, and will be continued as the results are inconclusive. Chas. Blanc

Hydrogenation of naphthalene. Horst Brückner. *Chem.-Ztg.* 55, 401-2 (1931).—A review. F. de Leeuw

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CA

The action of magnesium on dibromotoluene and dibromo-m-xylene. YU. S. ZAIKIND, S. KIRILLOVA AND V. NIKIFOROVA. *J. Gen. Chem.* (U. S. S. R.) 1, 103–8 (1931).—To a mixt. of 2.4 g. of Mg filings (previously washed with alc. and Et_2O and dried at 110°), a little dry Et_2O and a crystal of I was slowly added. 12.5 g. of 1,3,4-$C_6H_3MeBr_2$ (I) in 2 vols. of dry Et_2O and refluxed 35 hrs. in the water bath. On decompn. with ice water, the reaction mass was treated with 50 cc. of 0.8% H_2SO_4 to dissolve unchanged Mg, and the H_2 was measured, showing that 82.7% of Mg (and of Br) entered into reaction. The reaction mixt. was extd. with Et_2O and distd., giving 9 g. of resinous matter, 1.5 g. of PhMe, 3.5 g. of C_6H_4MeBr and 1.5 g. of high-boiling products. The tests showed that both Br atoms in I entered into the reaction with Mg, though incompletely and very slowly as compared with $C_6H_4Br_2$. Under similar conditions $C_6H_3MeBr_2$ does not react, with Mg and is completely recovered unchanged. When a mixt. of 2.4 g. of Mg filings, 13.2 g. of 1,3,4,6-$C_6H_2Me_2Br_2$, 3 vols. of dry Et_2O and a crystal of I are refluxed 60 hrs. and treated as described above, the reaction results in the consumption of 49.6% of Br with only 1 Br atom entering the reaction with Mg. Thus with increased no. of Me groups in the ring the reactivity of Br toward Mg is decreased. CHAS. BLAND

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Tribromonaphthalene obtained by bromination of naphthalene. Yu. S. Zalkind and M. V. Belikova. *J. Gen. Chem.* (U. S. S. R.) 1, 430-6(1931); cf. *C. A.* 25, 3985. Chas. Blanc

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10

Camphene. Yu. S. Zal'kind and M. G. Gurchikov. Russ. 28,007, Mar. 4 1932. HCl is split off from pinene-HCl by means of salts of naphthenic acid in the presence of an excess of caustic or without it as well as in the presence or absence of catalysts.

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The preparation of diethyl carbonate and its uses. Ya. S. Zal'kind and B. N. Rostovskii. *Plasticheskie Massui* 1932, No. 2-4, 6-11.—A critical review.
H. M. Leicester

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Derivatives of phenanthrene. Yu. S. Zalkind. *Anilinokrasochnaya Prom.* 2, 5-6, 7-10(1932).—Poor yields were obtained in all steps leading to diazo compds. of phenanthrene (I). I was nitrated by introducing it into concd. HNO_3, the mixt. of isomeric nitro compds. was reduced with H_2S and aq. NH_3 in 95% alc., the mixt. of NH_2 compds. was diazotized and coupled with 1,4-$C_6H_4(OH)SO_3H$, producing a dye which gave red dyeings on wool and silk. The work is being continued. C. B.

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Recent advances in the field of the technical use of catalysis. YU. S. ZAL'KIND, H. M. LEICESTER
J. Chem. Ind. (Moscow) 1932, No. 2, 19–26.—Review.

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Addition of hydrogen to acetylene derivatives. XIX. Relation between the direction and progress of catalytic hydrogenation and the nature of catalyst. Yu. S. Zalkind, M. N. Vishnyakov and L. N. Morev. *J. Gen. Chem.* (U. S. S. R.) 3, 91-113(1933); cf. *C. A.* 25, 2710. — While the hydrogenation of acetylene γ-glycols (I) in the presence of Pt black progresses uniformly with the formation of satd. glycols, the reaction with colloidal Pd proceeds in 2 stages: I rapidly adds 2 H atoms and then very slowly 2 more with the formation of satd. glycols. Such a sharp break in the process of hydrogenation is displayed only by γ-glycols. Acetylene alcs. combine with 4 H atoms without retardation in the reaction, while acetylene hydrocarbons (II), such as PhC : CH, add the 2nd pair of H atoms even faster than the 1st (*C. A.* 8, 1419; 10, 1355; 17, 1453). The hydrogenation of diphenylbutynediol acetate (III) also proceeds in 2 stages with retardation of the reaction after addn. of 4 H atoms followed by highly increasing acceleration of the reduction with the addn. of 4 more H atoms and the formation of diphenylbutane. These facts indicate that the course of hydrogenation is detd. not only by the speed, but that the reaction can proceed selectively, *viz.*, until the mols. of II (PhC : CH) or those of an unsatd. glycol ester are present in the reaction system they will react with H_2 prior to other mols. (styrene or satd. glycol ester) available in the mixt., though separately the latter could combine with H_2 with a greater speed. Such a selective action of Pd was also demonstrated by Bourguel (*C. A.* 22, 2099; 27, 2372), who concluded that the selective action of colloidal Pd suspended in starch always leads to hydrogenation of all available mols. of acetylene derivs. first to ethylene derivs. and thereafter to the satd. compds. This sp. action of Pd suspended in starch is not characteristic of other catalysts, such as Pt, or of Pd suspended in other protective colloids (*Z.* and Vilenkina, *C. A.* 10, 1466). Thus if the selective action of a catalyst is more definitely pronounced in some cases, it was of interest to inquire whether all acetylene derivs. act similarly, regardless of their structure, and to what extent this selective action is related to the chem. nature of the catalyst and is conditioned by the phys. form of the catalyst or the kind of protective colloid used for its suspension. By catalytic hydrogenation of acetylene derivs. there are formed *cis-* and *trans*-isomers of ethylene compds. While commonly the chief products are maleoid isomers, the *tetramethylbutynediol* (IV) and *tetraphenylbutynediol* produced also fumaroid isomers in various proportions, depending on the conditions of the reaction (*Z., J. Russ. phys.-chem. Soc.* 48, 1830(1916); Z. and Teterin, *C. A.* 26, 78; Z., *C. A.* 21, 2459). Bourguel (*C. A.* 19, 2551; 24, 2451) ascribed the formation of the 2nd isomer of ethylene glycol to some stereoisomerizing agents, such as protective colloids (protalbinic acid). IV, $Me_2C_6H_4$-C : COH and PhC : CH were hydrogenated in the presence

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of Pd and Pt in the form of colloidal suspensions in protective colloids, powd. blacks and with various carriers. As protective colloids were used starch, gum arabic, gum tragacanth, agar, Na protalbinate and casein for Pd, and starch and gum arabic for Pt. The tabulated results of the exptl. work led to the following conclusions. The general course of hydrogenation of acetylene deriv. is conditioned by the chem. nature of the catalyst and the structure of the hydrogenated compd., and while different protective colloids and catalyst carriers may be unlike in their activity, they exert but little influence on the general progress of hydrogenation. The results substantiate the chem. character of the mechanism of catalysis. The catalyst displays selective affinity for hydrogenated mols., showing in general a preference for mols. with a triple bond. The character of catalytic distribution between acetylene and ethylene mols. is conditioned by the nature of the catalyst and the structure of the compd. Thus Pd acts with more definite selectiveness than Pt, while PhC ≡ CH and styrene differ in their relation to a catalyst considerably more than acetylene alcs. from the corresponding ethylene alcs. All catalysts produced some α-tetramethylbutanediol by hydrogenation of IV in AcOEt or alc. soln. The yields of the α-isomer are independent of the presence of an amino acid (glutalbinic acid), and become greater with an increased quantity of a catalyst and a reduced time of addn. of H_2.

Chas. Blanc

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Preparation of unsymmetric γ-glycols of the acetylene series. I. Yu. S. Zalkind and V. K. Teterin. *J. Gen. Chem.* (U. S. S. R.) 3, 369-72(1933).—See *C. A.* 27, 3079.

C. Blanc

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Addition of hydrogen to acetylene derivatives. XIX.

1 Yu. S. Zalkind, V. K. Teterin and A. Ivanova. *J. Gen. Chem.* (U. S. S. R.) 3, 373-6(1933); cf. *C. A.* 27, 2679; 28, [illegible]

C. Blanc

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Preparation of unsymmetric γ-glycols of the acetylene series. II. Yu. S. Zalkind and A. I. Zakharova. *J. Gen. Chem.* (U. S. S. R.) 3, 432-3(1933); cf. *C. A.* 28, 2321[9].—See *C. A.* 27, 2079. W. P. Ericks

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CA

The addition of hydrogen to acetylene derivatives. XXI. The synthesis of symmetrical diphenyldi-α-naphthylbutanediol and its hydrogenation. Yu. S. Zalkind and S. V. Nedzvetzkii. *J. Gen. Chem.* (U. S. S. R.) 3, 872-7(1933).—A warm satd. soln. of 35 g. α-$C_{10}H_7Br$ (I) in Et_2O, added with stirring to $(BrMgC_6H_5)_2$ prepd. from 6 g. Mg, 30 g. Et_2O and C_6H_5Br gave after standing 20 hrs., refluxing 5 hrs. on a H_2O bath and standing 20 hrs. more, a mixt. which sepd. into 2 layers. The Et_2O layer with ice H_2O and dil. AcOH yielded 1.5 g. I. The heavy lower layer by extn. with Et_2O, drying with fused K_2CO_3, distn. of the Et_2O, and recrystn. successively from PhH and EtOH, gave 17 g. of sym. *diphenyldi-α-naphthylbutanediol* (II) $[Ph(C_{10}H_7)C(OH)C_6H_5]_2$, with 1 mol. of EtOH of crystn. Removal of the EtOH by heating at 125° for 2.5 hrs. gave II, m. 182-3°. II adds Br, does not react with $KMnO_4$ soln. in the cold, and gives with concd. H_2SO_4 an intense green coloration destroyed by diln. with H_2O. II (0.8 g.), heated at 140° with powd. KOH in H_2, gave I and C_6H_6, the latter identified by passing into NH_3-CuCl, forming C_6Cu_6. Only 1 optical isomer of II was obtained. II is hydrogenated more slowly than $[Ph_2C(OH)C_6H_5]_2$ (III) in EtOH with colloidal Pd, 0.005 mol. II with 10 mg. Pd requiring 5 hrs. for addn. of 2 H, 0.005 mol. III, 1.5 hrs. After addn. of 2 H, H_2 is much more slowly absorbed. II gives 2 geometric isomers, $[Ph(C_{10}H_7)C(OH)CH_2]_2$, 96% of *α-1,4-di-α-naphthyl-1,4-diphenylbutanediol* (IV) and 4% of the β-compd. (V). IV is difficultly sol. in EtOH and crystallizes out during the hydrogenation. IV seps. from EtOH with 1 mol. EtOH of crystn., m. 122-4° (decompn.) The EtOH-free IV thus formed or crystd. from petr. ether, m. 167°. IV adds Br more slowly than II, does not react with $KMnO_4$ in the cold and gives with concd. H_2SO_4 an intense green coloration changing rapidly to red and destroyed by diln. with H_2O. Oxidation of IV with $KMnO_4$ in AcMe with or without heating gave only CO_2. IV treated with CrO_3 in glacial AcOH gave an unidentified substance, yellow crystals, m. 163-3°. IV was the *cis*-compd. since it gave *2,5-di-α-naphthyl-2,5-diphenyl-3,5-dihydrofuran* (VI) when warmed for 1 hr. with 10 cc. Ac_2O and fused AcONa. VI was isolated from the reaction mixt. by pptg. the oil by addn. of H_2O, washing with H_2O, dissolving in PhH, and pptg. with petr. ether, crystals, m. 221-2° from EtOH. VI reacts with Br but not with cold $KMnO_4$. IV (1.3 g.) in AcOEt with 1.8 g. Pt added 2 H after 8.5 hrs. giving *1,4-di-α-naphthyl-1,4-diphenylbutanediol* (VII), m. 230°, from PhH. Addn. of 4 vols. H_2O to the mother liquor from IV gave a ppt. of IV + V. Successive recrystn. from PhH and EtOH gave V, m. 231-2°, which in contrast to IV, did not crystallize with EtOH. V resembled IV in its behavior toward Br, $KMnO_4$ and concd. H_2SO_4. The slower the hydrogenation of II, the greater the yield of V. V (1 g. in 15 cc. AcOEt with H_2 and 0.7 g. Pt gave VII in 2 hrs. V is the *trans*-isomer. The solubilities in 100 cc. PhH (temp. not given) were II 2.13 g., IV 0.26 g., V 0.90 g., VII 0.40 g., VI undetd. but very sol. VII was also prepd. from II in Et_2O by addn. of H_2 in the presence of Pt black.

Lewis W. Butz

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ZALKIND, Yu. S.

Action of bromine on α-naphthalenesulfonic acid. Yu. S. Zalkind, L. Veisbrut and M. Alekseeva. *J. Gen. Chem.* (U. S. S. R.) 3, 802-5(1933).—During the action of Br on α-$C_{10}H_7SO_3H$ there are formed 3 products: 5,1-$C_{10}H_6BrSO_3H$ and 1,4- and 1,5-$C_{10}H_6Br_2$. R. S.

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The action of acetylene-containing gases of cracked petroleum on copper and brass. Yu. S. Zalkind and B. A. Yovsi. *J. Applied Chem.* (U. S. S. R.) 6, 677-80 (1933). The expts. were carried out with cracked gases contg. up to 1.08% C_2H_2 at room temp., 50° and 80° and under atm. pressure and at 35 atm. The gas was passed for about one month at a velocity of 1.1 cu. m. in 24 hrs. over the metals, which were then analyzed for Cu acetylide. Only traces were found (up to 0.00145 g. on 100 cc. of Cu strips at 50°, and 0.003% on the brass strips). The attack on the metals at 80° was still less. A. A. B.

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Catalytic oxidation of benzene to maleic acid. Yu. S. Zalkind and S. Zolotarev. *J. Applied Chem.* (U. S. S. R.) 6, 681-4(1933).—The oxidation of C_6H_6 with air was effected in the presence of V_2O_5 pptd. on pumice stone or asbestos, by passing the air through C_6H_6 at 15°, preheating the mixt. up to 360° and effecting the oxidation at 410-30°. This procedure produced the highest yields, i. e., 14-17% of maleic acid calcd. on the C_6H_6 used. The unchanged C_6H_6 was adsorbed by cooled silica gel. Benzoquinone is formed as an intermediate product, because the oxidation of C_6H_6 takes place probably via PhOH, hydroquinone, quinone to maleic acid. The mechs. are described. A. A. Boehtlingk

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CA

Methylphenylcarbinol. Yu. S. Zalkind, M. K. Amusin and I. P. Berkovich. Russ. 34,547, Feb. 28, 1934. Ph. $CHClMe_2$ is hydrolyzed by heating with aq. soln. alkali carbonate.

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(A)

Esters of phosphorous acid. Yu. S. Zalikind and E. G. Dmitrieva. Russ. 34,548, Feb. 28, 1934. Al alcoholates are treated with PCl_3.

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Phenol esters of phosphorous acid. Yu. S. Zalkind and R. O. Dmitrieva. Russ. 34,555, Feb. 28, 1934. Phenols are heated with PCl_3 in the presence of $MgCl_2$.

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CA

Tetrachlorophthalic acid. Yu. S. Zal'kind and M. V. Belikova. Russ. 35,188, Mar. 31, 1934. Phthalic acid chloride is treated with Cl in the presence of Fe first at 120°, then at 170°. The product is worked up in the usual manner.

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Methyl(chlorophenyl)carbinol. Yu. S. Zal'kind, M. K. Antusin and I. P. Berkovich. Russ. 38,630, Sept. 30, 1934. $ClC_6H_4CHClCH_3$ is hydrolyzed by heating with alkali carbonate or the like.

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Chlorostyrene. Yu. S. Zal'kind, M. K. Amusin and I. P. Berkovich. Russ. 38,638, Sept. 30, 1934. Methyl-(chlorophenyl)carbinol is treated with a dehydrating agent.

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Tetrachlorophthalic acid. Yu. S. Zel'kind, M. V. Belikova and O. N. Simonova. Russ. 39,701, Nov. 30, 1934. Phthalic anhydride is chlorinated in the presence of Fe as catalyst.

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CA

The synthesis of styrene. Yu. S. Zal'kind, I. P. Berkovich and M. K. Amusin. *Plasticheskie Massy* 1934, No. 1, 14-20.—During 4-5 hrs. EtCl is added to a 3-fold excess of C_6H_6 contg. freshly prepd. $AlCl_3$ at 70°. After boiling 1 hr. more, 75% of PhEt is obtained. The more highly ethylated compds. which are also formed may be converted to PhEt by boiling with double their wt. of C_6H_6 and 10% of $AlCl_3$. Into the PhEt contg. 1% of $FeCl_3$ is passed Cl_2 at 70°, until 60% of the theoretical amt. has been added. After 2 hrs. refluxing, 60% of PhCH-ClMe (I) is obtained. When boiled for 20-30 min. with a small excess of quinoline, I gives 75-80% of styrene. A cheaper process is the hydrolysis of I by boiling it with a 25% excess of a 10-5% soda soln. This gives 90% of PhCH(OH)Me, which when dehydrated by heating with 3-5% of $KHSO_4$ yields 75% of styrene. App. for this

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Syntheses by means of vinylacetylene. Yu. S. Zal'kind and V. M. Pletz. *Trans. Leningrad. Chem.-Tech. Inst.* 1, 57-60(1934).—The Grignard reagent from 12 g. Mg and 55 g. EtBr cooled in a $CaCl_2$-snow mixt. reacted in 4 hrs. with 25 g. vinylacetylene in 75 cc. dry ether to give a sol. product vinylacetylenylmagnesium bromide (I). Twenty g. acetone was added, the reaction flask allowed to warm, and finally heated on a steam bath. After decompn. with dil. H_2SO_4, washing, drying over potash and distn. *in vacuo*, 30-5 g. (32% of theory) of 2-methyl-5-hexen-3-in-2-ol (II), $H_2C{:}CHC{:}CC(OH)Me_2$, b. 75°, b_{10} 64° resulted. II is a yellow liquid, insol. in water, sol. in org. solvents, absorbs Br_2 and darkens with H_2SO_4. Addn. of 20 g. of dry AcOEt to I, stirring for 2 hrs., decompn. with ice-cold dil. H_2SO_4 in N_2, drying over $CaCl_2$ and distg. at 8 mm. at 110° gave 5 g. of 5-methylnona-1,8-dien-3,6-diin-5-ol (III), $HOCMe(C{:}CCH{:}CH_2)_2$. The mol. wt. in benzene was 143. On heating at 25 mm. III decomposes and at 1 atm. it explodes. III is insol. in H_2O, absorbs Br_2 to give an oily bromide, and at 50° reacts with anhyd. oxalic acid to give an orange-red hydrocarbon. Also in *J. Gen. Chem.* (U. S. S. R.) 4, 1088-90 (1934).

F. H. Rathmann

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PROCESSES AND PROPERTIES INDEX

Esters of hydrogenated phenols. Yu. S. Zal'kind and M. O. Gerchikov. *Plasticheskie Massy* 1938, No. 3, 37-40.--Cyclohexanol (I), gently heated with 10% excess AcOH and 0.3% H_2SO_4 for 0.5 hr., yields 83% of the ester. AcOH as weak as 60% may be used. Under the same conditions methylcyclohexanol gives 78-80% of the corresponding ester. I and 85% HCO$_2$H, allowed to stand in the cold without a catalyst for 1 day, give 85-93% of the formate. I and anhyd. $(CO_2H)_2$, refluxed without a catalyst, give the diester, but the monoester cannot be so obtained. H. M. Leicester

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

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The preparation of triphenyl and tritolyl phosphates. Ya. S. Zal'kind and E. G. Dmitrieva. *Plasticheskie Massy*, 1936, No. 4, 28-30.—The phosphates are best prepd. from PhOH or MeC_6H_4OH and $POCl_3$ in the presence of MgO as a catalyst. H. M. Leicester

COMMON ELEMENTS

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The nitration of β-bromonaphthalene. Yu. Zal'kind and F. Filinov. *J. Gen. Chem.* (U. S. S. R.) 4, 979-81 (1934).—The yield of β-bromonaphthalene (I) obtained from β-$C_{10}H_7NH_2$ by the method of Chichibabin (*C. A.* 7, 3599) can be increased to 42% as follows: Add the diazo compd. in very small portions with energetic stirring to freshly prepd. CuBr and HBr at 70-80°. I stirred energetically with HNO_3 (d. 1.4) at 25-35° for 6 hrs. gave 10% of a *nitrobromonaphthalene* (II), m. 110-11° from hot 80% AcOH. It was necessary to sep. the 1st crystals quickly from the still warm soln. as they were quickly followed by a flocculent material m. 75-105°. Since the 3 known nitro-β-bromonaphthalenes (1,2, 3,2 and 4,2), m. 102-3°, 83-3° and 97-8°, resp., II must be nitrated in the 2nd ring. II heated with HBr in a sealed tube gave 1,7-dibromonaphthalene, indicating that II is probably the 1,7-isomer. A small quantity of the 1,2-isomer was isolated from the mother liquor from II. Lewis W. Butz

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Action of bromine on β-naphthalenesulfonic acid. Yu. P. Zal'kind and M. V. Belikova. *J Gen. Chem.* (U. S. S. R.) 4, 1211-16(1934); cf. *C. A.* 28, 6130[illegible]. By the action of Br on β-$C_{10}H_7SO_3H$ in an equal part of ice-cold H_2O there is formed a mixt. of unreacted $C_{10}H_7SO_3H$, $Br_2C_{10}H_5SO_3H$ and equal parts of 5,2- and 8,2-$BrC_{10}H_6SO_3H$. Chas. Blanc

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Amines of phenanthrene series. Yu. S. Zelkind and I. Ya. Chaikin. Russ. 40,088, Jan. 31, 1935. Phenanthrols are heated with an excess of $(NH_4)_2SO_4$ and NH_3 to 140-190°; phenanthrylamines are sepd. from the reaction mixt. by soln. in acid and a reppta. with alkali.

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Camphor. Yu. S. Zal'kind, Yu. L. Mikhelis and M. A. Styalov. Russ. 42,076, Mar. 31, 1935. Camphor is prepd. by dehydrogenating borneol or isoborneol by heating in the presence of $3CuO.2CO_2.H_2O$.

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Methods of synthesizing camphor from Soviet turpentine. Yu. S. Zal'kind, E. N. Rostovskii, I. A. Arbuzova and V. K. Teterin. *Narodnyi Komissariat Tyazkoi Prom. S. S. S. R., Leningrad, Plastmassy* 1, 589–603 (1935).—Various synthetic methods are compared. Drying pinene with CaO does not improve the yields of pinene-HCl from it. Salts of naphthenic or abietic acids are better than oleates in the formation of camphene from pinene-HCl. Oxidation of camphene to camphor goes best with a soln. of 50–90 g. of $Na_2Cr_2O_7$ per l. treated with a 25% excess of H_2SO_4. The addn. of small amts. of C_6H_6 or benzine during oxidation raises the yield of camphor. The use of Pb app. slows the reaction. For the prepn. of isoborneol from pinene the best acid to use is tetrachlorophthalic acid. H. M. Leicester

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Investigations in the field of vitamin A synthesis. Yu. S. Zal'kind, S. Zouis and N. Blokhin. *Compt. rend. acad. sci. U. R. S. S.* 2, 57-60(in German 61-3)(1935).—β-Ionone and the Mg deriv. of vinylacetylene were heated on a H_2O bath for 2 days till the odor of ionone disappeared. After the usual treatment a viscous liquid, *3-methyl-1-(2',6',6'-trimethyl-1'-cyclohexenyl)-1,6-heptadien-4-ine 3-ol* (I), $Me_3C.(CH_2)_2.CMe:CCH:CHCMe(OH)C:CCH:CH_2$, $b_{0.1}$ 135-45°, d_{20} 0.9818, n_D^{20} 1.5284, was obtained. This was reduced by almost the calcd. vol. of H_2 and a Pd catalyst to *β-(2,2,6-trimethylcyclohexyl)ethylmethylbutylcarbinol*, $Me_3C.(CH_2)_2.CHMe.CH(CH_2)_2CMe(OH)(CH_2)_2.CH_2$, $b_{0.1}$ 125-9°, which was identified by oxidation with $K_2Cr_2O_7$. This gave tetrahydroionone (II) and $PrCO_2H$. The latter appeared to be contaminated with a little trimethylcyclohexylacetic acid. II was identified by comparison with *tetrahydroionone*, $b_{0.1}$ 112-15°, d_4^{20} 0.9303, d_4^{20} 0.9224, n_D^{20} 1.4766 (*semicarbazone*, m. 165-6.5°), obtained by hydrogenating ionone with H_2 and Pd. While I had many of the chem. characteristics of vitamin A, tests on rats showed that it was not a vitamin.

John E. Milbery

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Syntheses of solvents and plasticizers. Yu. S. Zal'-kind. *Trans. VI Mendeleev Congr. Theoret. Applied Chem.* 1932 2, Pt. 1, 729-30(1935). A review on the synthesis of methylcyclohexane, cellosolve, ethyl ether, camphor, phthalates, phosphoric acid esters, etc.
B. R. Stefanowsky

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1ST AND 2ND ORDERS

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3RD AND 4TH ORDERS

The catalytic dehydrogenation of ethylbenzene to styrene. Ya. S. [illegible]kind and G. L. Balavskii. *Plasticheskie Massy* 1935, No. 3, 9-12.—PhEt mixed with N is passed over a catalyst of 10% ZnO and 90% Al_2O_3 at 10-13 mm. pressure, 60-70°, and a rate of [illegible] g. per min. to yield 89.74% styrene.

H. M. Leicester

COMMON ELEMENTS

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The addition of hydrogen to acetylene derivatives. XXII. Bis(1-hydroxycyclohexyl)acetylene, and its derivatives. Yu. S. Zal'kind. *J. Gen. Chem.* (U. S. S. R.) 5, 1723-7(1935); cf. *C. A.* 28, 2707[8].—Previous study of the rate of addn. of H_2 to acetylene γ-glycols showed that biternary glycols are hydrogenated more slowly than bi-secondary glycols (Z. and Neishtab, *J. Russ. Phys.-Chem. Soc.* 50, 36(1918)), and that the reaction velocity decreases with the increasing mol. wt. of the radicals (cf. Z. and Buistryakov, *J. Russ. Phys.-Chem. Soc.* 47, 680 (1915); Z. and Markaryan, *C. A.* 11, 584). The influence of cyclic radicals, connected directly with the HO-bearing C, on the rate of hydrogenation of biternary acetylene glycols was studied by hydrogenating 0.01 mol. of *bis(1-hydroxycyclohexyl)acetylene* (I) in 50 cc. of alc. in the presence of various amts. of Paal's colloidal Pd at 18-21° and 756-61 mm. pressure. I, bs 183° (decompn.), m. 109° (103°, Dupont, *C. A.* 8, 1579), was prepd. by the Dupont method. I gives with AcO and KOAc the diacetate, m. 45.5-6.5°, and with Br_2 $CH_2(CH_2)_4C(OH)CBr:CBrC(OH)(CH_2)_4CH_2$ m. 101.5-7.5°. Like other biternary glycols, it easily adds 2 H atoms, after which the rate of reaction declines. I differs from other glycols with radicals of nearly equal or even smaller magnitude. It is more easily hydrogenated than $(MeEtC(OH)C)_2$ (II) and $(Et_2C(OH)C)_2$ (III). The rate of hydrogenation of $(Me_2C(OH)C)_2$, II and III gradually decreases with increasing mol. wt. of the radicals (15, 55 and 145 min., resp.) (*loc. cit.*), while that of I (30 min.) shows no relation to other glycols. The probable stereochem. causes influencing the reaction are being investigated. With increasing ratios of the catalyst (1, 2, 4, 8 and 10), the hydrogenation velocity at first increases at a disproportionate rate and then imperceptibly drops (1, 5.6, 20.9, 29.4 and 47.8, resp.). Under all conditions of hydrogenation only *s-bis(1-hydroxycyclohexyl)ethylene*, m. 152°, is obtained. I is difficultly oxidized, giving only 0.5 g. adipic acid on oxidizing 5 g. of the glycol with 5.24 g. $KMnO_4$ (1% soln.) at room temp. for 12 hrs. XXII. Dihydroxybutanediol and its hydrogenation. Yu. S. Zal'kind and V. I. Tzereshko. *Ibid.* 1708-71.—The effect of substitution of an open-chain for a Ph radical of an equal no. of C atoms in an acetylene glycol on the rate of addn. of

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H_2 was studied by a comparative hydrogenation of *dihexylbutinediol* (I) and diphenylbutinediol (II) (cf. Z. and Neishtab, *loc. cit.*). I, m. 47-9°, was obtained in 99% yield by the method of Iotzich (*J. Russ. Phys.-Chem. Soc.* 38, 656(1906)) and Dupont (*loc. cit.*). I after repeated crysta. from ligroin sepd. a few crystals of a compd., m. 68-70°. The mol. wt. of I is 251 (Me_2CO) and 510 (C_6H_6) (calcd., 254.2). I was hydrogenated in the presence of colloidal Pd as described above. The reaction was completed in 15 hrs., requiring an addnl. introduction of Pd after 6 hrs. of hydrogenation. A break in the rate of hydrogenation after the addn. of the 1st 2 H atoms is sharply defined. With 1 mg. and 7.5 mg. of the catalyst the relative addn. of H_2 for the 1st 5 min. is, resp., 12.76 and 65.16% for I, 7.8% for II and 30.44 and 99.4% for dimethylbutinediol. Thus a substitution of hexyl for Ph, but not for a lighter radical, considerably accelerates the hydrogenation. I on the addn. of 2 H atoms gives 2 ethylene glycols, $[Me(CH_2)_5CH(OH)CH:]_2$, cryst., m. 44-5°, and liquid, b_{14} 195-200°, d_4^{20} 0.84. The cause of the formation of 2 ethylene glycols may be that I is a mixt. of *d* + *l* and *i*-isomer, or that in the hydrogenation of I there are formed 2 geometrical isomers corresponding to the same acetylene glycol. The same correlation of the reaction products obtained by the rapid hydrogenation (8-10 mg. Pd) and by the *slow* hydrogenation (1 mg. Pd) disproves the 2nd postulation. Furthermore in case of geometrical isomerism, an exhaustive hydrogenation of the 2 ethylene glycols in the presence of Pt black catalyst would give identical satd. glycols, because the asym. C atoms are not affected in hydrogenation. I on the addn. of 4 H atoms gave 2 products, m. 105-6° and m. 85-6°, analyzing for hexadecane-7,10-diol. Exhaustive hydrogenation of the cryst. ethylene glycol gave only 1 satd. glycol, m. 85-6°, while that of the liquid ethylene glycol gave 2 isomers in the ratio of 1:1. It follows that the 2 ethylene glycols are not geometrical isomers but are derived from the 2 isomeric I. I, treated with Br_2 in $CHCl_3$, gave 45% of dibromide, m. 97-8° (Dupont, *loc. cit.*). This on cleaving Br_2 with Zn dust in alc. gave I, m. 53-4°, which on hydrogenation with colloidal Pd gave only the cryst. ethylene glycol, m. 44-5°, and with Pt black the satd. glycol, m. 85-6°.

Chas. Blanc

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1ST AND 2ND ORDERS

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B. V. Bulzov. Yu. S. Zal'kind. *J. Applied Chem.* (U. S. S. R.) 8, 742 56(1935). Obituary and a review of B.'s research with 68 references. A. A. B.

3RD AND 4TH ORDERS

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Production of tetrachlorophthalic acid. Yu. S. Zel'kind and M. Belikova. *J. Applied Chem.* (U. S. S. R.) 8, 1210-13 (in German 1213) (1935).—A yield of 90-5% $C_6Cl_4(CO_2H)_2.H_2O$ (I), m. 244-5° (open tube), is obtained by chlorinating $C_6H_4(CO_2H)_2$ (II) in the presence of 3% Fe filings at 240-50° to the correct m. p. of the reaction product. The chlorination without stirring proceeds slowly, requiring 63-5 hrs. for 300 g. and 110 hrs. for 2 kg. II. $C_6H_4(COCl)_2$ reacts with Cl more rapidly, giving 98% I. I loses the water of crystn. at 100° and the water of constitution at 110°, giving $C_6Cl_4(CO)_2O$.

Chas. Blanc

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α-Vinylnaphthalene and some of its derivatives. Yu. S. Zal'kind and S. A. Zonis. *J. Gen. Chem.* (U. S. S. R.) 6, 988-98 (1936).—The polymerization ability of high-mol. unsatd. compds. in relation to their structure and the properties of the resulting polymers was studied in the case of α-vinylnaphthalene (I) and its derivs. substituted in the side chain. I, RCH:CHMe (II), RCMe:CH_2 (III), RCMe:CHMe (IV), and RCEt:CHMe (V) (R = $C_{10}H_7$), were prepd. by the Grignard reaction by interaction of $C_{10}H_7MgBr$ and the corresponding alcs. with subsequent dehydration of the resulting alcs. with $KHSO_4$ (cf. Shorygin and Shorygin, *C. A.* 29, 1985[9]). The alcs. were identified as the acid esters of $C_6H_4(CO_2H)_2$. The polymerization was effected without catalysts and in the presence of BrO_2H and floridin at 20°, 40°, 60° and at higher temps. The polymerization rate was detd. either by vacuum distn. of the monomer and weighing the polymer residue, or by measuring the relative viscosity in the Ostwald viscometer. I under all conditions gave only a polymer and in the amt. proportional to the elapsed time. The viscosity of polymerization product increased at a greater rate than the percentage of polymer in the reaction mixt. Thus, viscosities of 9.81 and 30.28 with 10.28% and 21.63% polymer resulted after 11 and 22 days, resp. The viscosity of a polymer at an equal concn. obtained at 20° is greater than that of the product obtained at 40°. The resistance to heat is 150-60° for I polymerized at 20°, and 110-20° for the product obtained at 20°. The polymer of I obtained in the presence of BrO_2H catalyst showed poor insulating properties and considerable dielec. losses. The derivs. of I showed poor polymerizing abilities even at elevated temps. (boiling water bath). V failed to polymerize under all conditions. III and IV showed no increase in the viscosities after 44 days of polymerization in the presence of 0.5% BrO_2H, while II gave 8.2% polymer. III and IV doubled the viscosities in the presence of floridin after 100 days at 20°. The substituted I gave chiefly dimers and no polymers as in the case of I. The sym. derivs. of I are more easily polymerized than the asym. ones. I, b_4 105-6°, d_4^{20} 1.0774, n_D^{20} 1.6385, M. R. 52 (50.41 calcd.), exaltation 1.60. II, b_4 112-13°, d_4^{20} 1.0429, d_4^{40} 1.0283, n_D^{20} 1.6335, exaltation 2.51, M. R. 55.03 (57.54 calcd.). III, b_4 112-13°, d_4^{20} 1.027, n_D^{20} 1.6142. IV, b_4 113.5-15°, d_4^{20} 1.0533, n_D^{20} 1.6137, exaltation 0.4, M. R. 60.05 (59.45 calcd.). V, b_4 119-20°, d_4^{20} 1.034, n_D^{20} 1.597, exaltation 0.35, M. R. 64.03 (64.27 calcd.). More than 30 references. Chas. Blanc

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Adipic acid. Yu. S. Zal'kind and I. F. Markov. Russ. 54,391, Jan. 31, 1937. Adipic acid is prepd. by oxidizing cyclohexene with soln. of $K_2Cr_2O_7$ in the presence of H_2SO_4.

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Cyclohexanol. Yu. S. Zal'kind and I. F. Markov. Russ. 51,870, Sept. 30, 1937. H_2SO_4 (70-80%) is added to cyclohexene in the absence of a catalyst at such a rate as to keep the reacting mass at room temp.

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Unsaturated alcohols. Vu. B. Kal'kind and B. A. Zokis. Russ. 51,005, Oct. 31, 1937. Alcs. contg. at least one triple and two double bonds are prepd. from acetylene bivinylmagnesium halides by the action of aldehydes or ketones.

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Obtaining esters of dicarboxylic acids. Yu. S. Zal'kind and M. G. Gerchikov. *Narodnyi Komissariat Tyazhelot Prom. S. S. S. R., Nauch.-Issledovatel. Inst. Plasticheskikh Mass., Plasticheskie Massy, Sbornik 2,* 191–7(1937).—Yields of 85–95% of the following adipates are obtained by standard methods: dicyclohexyl, m. 37°; bis(methylcyclohexyl); di-Et; diisoamyl, b_{15} 190°, d_{20} 0.9455, n^{20} 1.4343; *diisobutyl*, b_{15-17} 186–8°, d_{15} 0.9543, n^{20} 1.4301; *di-Bu* b_{16-17} 190–1°, d_{20} 0.950, n^{20} 1.4339.

H. M. Leicester

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1ST AND 2ND CODES

PROCESSES AND PROPERTIES INDEX

The catalytic dehydrogenation of borneol and isoborneol. Yu. S. Zal'kind, Yu. L. Mikhelis, M. A. Stoyalov and R. L. Levin-Kogan. *Narodnyi Komissariat Tyazhelol Prom. S. S. S. R., Nauch.-Issledovatel. Inst. Plasticheskikh Mass., Plasticheskie Massy, Sbornik* 2, 263-81(1937). $Ni(OH)_2$ reduced with H_2 at 250-300° needs no activator for liquid-phase dehydrogenation of borneol. Isoborneol is more difficult to dehydrogenate. $Cu_2(OH)_2CO_3$ is a cheaper and more stable catalyst. It should be mixed with $MgCO_3$ to increase the surface. Boiling the borneol for a short time with CaO or $Ca(OH)_2$ before reaction eliminates traces of acids which slow the dehydrogenation and makes the catalyst more active. H. M. Leicester

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The synthesis of camphor by the method of J. Ritter. Vu. S. Zal'kind and A. Egorova. *Narodnyi Komissariat Tyazkoi Prom. S. S. S. R., Nauch.-Issledovatel. Inst. Plasticheskikh Mass., Plasticheskie Massy, Sbornik 2,* 123-30(1937); cf. *C. A.* 27, 4780.—Minor modifications are suggested. H. M. Leicester

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Ch

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Obtaining adipic acid from oil distillates. Yu. S. Zal'kind and I. F. Markov. *Narodnyi Komissariat Tyazhelol Prom. S. S. S. R. Nauch.-Issledovatel. Inst. Plasticheskikh Mass., Plasticheskie Massy, Sbornik 2,* 431-44(1937). The cyclohexane obtained from the 80-110° fraction of an oil distillate can be oxidized directly to adipic acid by concd. HNO_3, but the yield is low. Continuous chlorination of the cyclohexane gives 90% cyclohexyl chloride which easily splits out HCl when passed over activated C with steam at 250-300° to give 92% cyclohexene. Oxidation of the latter with a mixt. of H_2SO_4 and $Na_2Cr_2O_7$ gives 45-49.5% adipic acid. H. M. Leicester

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A new synthesis of bisacetylene glycols. Yu. S. Zal'kind and M. A. Alzikovich. *J. Gen. Chem.* (U.S.S.R.) 7, 227-33(1937); cf. *C. A.* 30, 2932[4].—In a previous study of the condensation of monosubstituted acetylenes in the presence of CuCl + NH_4Cl acidified with HCl, it was found that PhC:CH gives diphenylbiacetylene. The application of this reaction to the synthesis of biacetylene derivs. of OH compds. was begun with tertiary acetylene alcs. The reaction leads to the formation of tertiary biacetylene glycols, according to the scheme: 2 RR'C(OH)C:CH → [RR'C(OH)C:C]$_2$ + H_2. Thus, Me_2C(OH)C:CH (I) gave [$Me_2C(OH)C:C]_2$ (II) and 1-hydroxycyclohexylacetylene (III) gave *bis-(1-hydroxycyclohexyl)biacetylene* (IV). I, b. 103.5-4°, d_4^{20} 0.8652, n_D^{20} 1.4202, M. R. 24.58 (calcd. 24.81) (cf. Favorskii, U. S. S. R. pat. 31,017), (2 g.) was introduced into 2 g. CuCl and 6 g. NH_4Cl in 25 cc. H_2O at room temp. The reaction was completed in 3-5 min., giving 100% II, m. 132-2.5° (C_4H_4). II in EtOAc hydrogenated in the presence of Pt black added 8 H atoms, forming 2,7-dimethyloctane-2,7-diol, m. 88-9° (ligroin), 57-9° (H_2O) (cf. Zelinskii, *J. Russ. Phys.-Chem. Soc.* 38, 931(1906)). The reaction proceeds without the usual addn. of HCl and gives equally good results in the absence of NH_4Cl and with NH_4OH soln. of Cu_2O. Since the reaction affords 100% II without the pptn. of Cu and evolution of a gas, it might be assumed that the atm. O_2 acts as an acceptor of the H_2 liberated in the reaction. In fact, the reaction in a H atm. proceeds very slowly, giving after 1 hr. but few crystals of II. In an atm. of pure O_2, the condensation takes place very rapidly with a decrease of the O_2 vol., corresponding to the amt. required for oxidizing CuCl (Cu_2O) and the H_2 liberated in the reaction. III, b. 99-101°, d_4^{20} 0.9668, n_D^{20} 1.4750, M. R. 36.17 (calcd. 36.47), obtained by the Rupe method (*C. A.* 22, 2928), was contaminated with about 8% cyclohexanol. IV, m. 173-4° (C_4H_4), resulted in nearly 100% yield from 3.1 g. III and 3 g. CuCl and 9 g. NH_4Cl in 30 cc. H_2O. The reaction is completed at room temp. in 20-5 min. in O and in 2-3 hrs. in air. In N an insignificant amt. of IV resulted after 12 hrs. of interaction. The condensation of III in the presence of Cu_2O in NH_4OH gave 62.5% IV. Hydrogenation of 2 g. IV with 2 g. Pt black resulted in the addn. of 22 H atoms and formation of α,δ-dicyclohexylbutane, b_{20} 169°, and with 0.6 g. Pt black in bis(1-dihydroxycyclohexyl)-1,4-butane. Chas. Blanc

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1 **The addition of hydrogen to acetylene compounds. XXVII. The catalytic hydrogenation of 2,5-dimethyl-1,5-hexadien-3-ine.** Yu. S. Zal'kind and Z. V. Sasagina. *J. Gen. Chem.* (U. S. S. R.) 7, 470-5(1937); cf. *C. A.* 31, 1012[5].—This compd. is best prepd. by dehydration of the corresponding glycol with H_3PO_4. When it is hydrogenated over Pt, the normal satd. hydrocarbon is produced, but if Pd is used, only 6 H atoms are absorbed. The first step is probably to CH_2:CMeCH:CHCMe:CH_2.

2 This then gives chiefly 2,5-dimethyl-1-hexene, though the product seems to contain not more than 3-5% of 2,5-dimethyl-3-hexene. H. M. Leicester

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The addition of hydrogen to acetylene derivatives XXVII. Hydrogenation of symmetrical diphenylditolylbutinediol. Yu. S. Zal'kind and E. R. Martinson. *J Gen. Chem.* (U. S. S. R.) 7, 815-17(1937); cf. *C. A.* 31, 4304[1].—Continuing the study on the hydrogenation of tetraarylbutinediols to give both possible geometric isomers of the olefin glycol, *1,4-diphenyl-1,4-di-p-tolylbutene-1,4-diol* (I) is synthesized and its behavior on hydrogenation shown to conform with that of previously studied tetraarylbutinediols. To BrMgC:CMgBr, prepd. from 8 g. Mg and 35 g. EtBr, is added 80 g. (15 g. excess) *p*-MeC$_6$H$_4$COPh (IV), m. 57°, prepd. by the Friedel-Crafts reaction from BzCl and PhMe, the reaction mixt. allowed to stand 5 days, decomp'd. with dil. AcOH, and the ppt. (49 g.) recrystd. twice from EtOH to give 25 g. (35.9%) I, m. 146°. The structure of I is proved by fusion with KOH in a stream of H to yield C$_2$H$_2$ and IV, m. 57°. I (2.09 g.) in dry Et$_2$O contg. Pt black (0.8 g.) is completely hydrogenated in 80 min. to *1,4-diphenyl-1,4-di-p-tolylbutane-1,4-diol* (II), crystals from acetone, m. 176°. I (2.09 g.) in 100 ml. EtOH contg. colloidal Pt (10-50 mg.), hydrogenates with some difficulty to give the 2 geometric isomers of *1,4-diphenyl-1,4-di-p-tolylbutene-1,4-diol* (III): trans form (19.8% yield), crystals from acetone or alc., m. 188-90°; cis form (70.7% yield), crystals from ligroin, m. 96°. Both forms of III, in the presence of Pt black, reduce to II. Concd. H$_2$SO$_4$ is an excellent reagent to distinguish between II and the 2 forms of III: II gives a green-yellow coloration, while both forms of III give a characteristic crimson color, passing rapidly through a series of intermediate colors to a red-green. J. L.

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Catalytic oxidation of some aromatic compounds

Yu. S. Zal'kind and V. V. Kesarev. *J. Gen. Chem.* (U. S. S. R.) 7, 879-81(1937); cf. C. A. 31, 6312[9]. The behavior of $C_{10}H_8$ derivs., pyridine (I), quinoline (II) and of carbazole (III), when admixed with air, or air enriched in O_2, and passed at 400-600° over V_2O_5 (90%)-U oxides (10%) catalyst, deposited on pumice, is studied and the stability of different nuclei detd., α-$BrC_{10}H_7$ oxidized at 400° gives only BzOH (IV) and traces of phthalic acid (V). α-$O_2NC_{10}H_7$, and α- and β-$H_2NC_{10}H_7$, at 420°, give only phthalimide and a small amt. of V. β-$HOC_{10}H_7$ at 420° gives only IV. These results indicate that the unsubstituted nucleus is more stable in all cases studied. I oxidized at 430-600° gives some unchanged I, the remainder completely decompg. to H_2O, CO_2, NH_3 and N oxides. II is oxidized with great difficulty. At 400° it remains unchanged, at 420-30° it behaves like I, while at 500°, with air contg. 10% O_2, it explodes. III remains unchanged at 400-500°, while at 600° it partially undergoes complete combustion, at times with explosive violence.

John Livak

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The addition of hydrogen to acetylene derivatives XXVIII. Bicyclohexenylacetylene and its hydrogenation. Yu. S. Zal'kind and N. N. Shuvalov. *J. Gen. Chem.* (U. S. S. R.) 7, 1235-45(1937); cf. *C. A.* 31, 5787[9].—*Bicyclohexenylacetone* (I) was prepd. in 80% yield by dehydration of dihydroxycyclohexylacetylene, m. 108.5-9°, with 40-5% of freshly fused $NaHSO_4$ at 145-55° for 2-2.5 hrs. I, b_{12} 158-9°, d_{20}^{20} 0.9904, n_D^{20} 1.15370, M. R. 61.87 (calcd. 50.52 for $C_{12}H_{18}$), exaltation 2.35, P 481.0 (calcd. 480.0). It is oxidized with $KMnO_4$ in Me_2CO to adipic acid. When I is hydrogenated over Pt black, it absorbs 8 H atoms, giving sym. dicyclohexylethane, $b_{12.5}$ 133-4°, d_{20}^{20} 0.8772, n_D^{20} 1.4737, M. R. 62.10 (calcd. 62.17), P 530.8 (calcd. 530.8). I hydrogenated over Pd absorbs only 6 H atoms, giving a compd. with 1 double bond and analyzing for $C_{12}H_{20}$, b_{12} 136-7°, d_{20}^{20} 0.9000, n_D^{20} 1.4910, M. R. 61.85 (calcd. 61.68), exaltation 0.17, P 513.9 (calcd. 513). It appears to be $C_6H_5CH_2CH_2C_6H_{11}$.

Chas. Blanc

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The addition of hydrogen to acetylene derivatives. XXIX. The action of sodium and alcohol on acetylene glycol and the geometric isomers of tetramethylbutenediol. Yu. S. Zal'kind and S. V. Bukhovets. *J. Gen. Chem.* (U. S. S. R.) 7, 2417-22(1937); cf. *C. A.* 31, 6208[6].—Bourguel and Rambaud (*C. A.* 24, 2720) claim to have obtained a *trans*-tetramethylbutenediol, m. 101-2° (I), from tetramethylbutynediol (II) by reduction with Na and EtOH, and that the compd. of Z. m. 75-6° (III) is an impure mixt. When II is reduced over Pt it adds 4 H atoms and gives tetramethylbutanediol. Over Pd it adds 2 H atoms to form III. When it is boiled with Na and iso-AmOH it forms C_2H_2, a very sensitive test for acetylene alcs. I forms the same dibromide that is obtained from II. It is therefore a dimorphic form of II. When melted II is seeded with I, the solid product is mostly I though the reverse change does not occur. III is actually the *trans*-tetramethylbutenediol.

H. M. Leicester

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CA

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Catalytic oxidation of phenanthrene by air. Ya. S. Zal'kind and V. V. Kesarev. *J. Applied Chem.* (U.S.S.R.) 10, 99-104(in German 104)(1937).—The oxidation of phenanthrene in the presence of pumice impregnated with 33% V_2O_5 at 400° (the temp. of the phenanthrene was 275°, which corresponds to 3% of phenanthrene in the gas mixt.) by O_2 and by air to which 12% of O_2 was added, yielded CO_2, H_2O, and only 2% of phthalic acid. Preheating phenanthrene to 225° and carrying the reaction out at 370° and a duration of contact of 4 sec., the yield of phthalic acid was highest (11.5%). Addn. of CO_2 to the air did not increase the yield of phthalic acid, but increased moisture content of the air yielded 14% of solid acids m. 177°, contg. mainly phthalic acid and some diphenic acid (m. 210°); further increase of the moisture (to 60% did not improve the yield. The best yield (21%) of acids was obtained by using pumice with a concn. of NH_4VO_3 equal to 25%. The gradual introduction of phenanthrene also increased the yield of acids; this can be explained by gradual increase of the activity of the catalyst. Pure WO_3 did not catalyze the reaction at all, and its mixt. with V (W 20 and V 80%) yielded only 12% of acids. The mixt. of Mo 10 and V 90% catalyzed the reaction, yielding 30% of acids, but pure Mo is a poor catalyst. The mixt. of U oxide and V_2O_5 (U 30 and V 70%) catalyzed the reaction to a greater extent, yielding 41% of solid acids. The optimal conditions for the last catalyst were 10 g. of the catalyst, a contact time of 1.2 sec., reaction temp. of 430° (if the phenanthrene was heated to 240°) or 440° (if it was heated to 215°). The last catalyst was very stable; after 12 expts. its activity had not decreased at all. The catalyst composed of V 60, Mo 10 and U 30%, at 350-80° (the phenanthrene was heated to 215°) and a contact time of 2 sec., yielded 30% of solid acids. Addn. of the oxides of Zn, Cu, Ag, Bi to the U-V catalyst had practically no influence on the yield. The acids obtained had in all cases a yellow color, m. 160°, and consisted of phthalic acid, which partially changed to BzOH; in some expts. a small yield of diphenic acid and phenanthrenequinone was obtained, and, in some, the acid products treated with H_2SO_4 gave a red color, characteristic of fluorenonecarboxylic acid, and the neutral products treated with concd. H_2SO_4 gave a violet color, characteristic of fluorenone. The oxidation of phenanthrene was also carried out in liquid phase, by passing a stream of the air or O_2 through the liquid phenanthrene (at 240°), but the yield was very poor. In this case, the oxidation with air yielded diphenic acid, and that with O_2 maleic acid. The mechanism of the oxidation of phenanthrene is given as follows: phenanthrene → phenanthrenequinone → 6,6'-diphenic acid → o-phthalic acid → BzOH → maleic acid → CO_2 + H_2O; and the formation of the fluorenenone and fluorenonecarboxylic acid can be explained by the following scheme: 6,6'-diphenic acid → 2,6'-diphenic acid → fluorenonecarboxylic acid → fluorenone → o-phthalic acid. Four references.

A. A. Podgorny

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PROCESSES AND PROPERTIES INDEX

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Preparation of the metacrylic acid ester from isoölleic acid. Yu. S. Zal'kind and I. F. Markov. *J. Applied Chem.* (U. S. S. R.) 10, 1042-4(in German 1044)(1937).—Isoölleic acid was chlorinated in the presence of elec. light (60-w. globe), starting the reaction at first at 50°; then the temp. could be dropped to 20-30° without diminishing the velocity of the reaction. The product was fractionated, yielding unchanged acid, α- and β-chloroisoölleic acid. Under the above conditions the yield of β-isomer was the greatest. The Me esters of both isomers were prepd. by heating the acids with 2 parts of MeOH in the presence of H_2SO_4 (concd.) for 3 hrs. The resulting mixt. were neutralized with soda (aq.) and the ester of the α-isomer was extd. with Et_2O, whereas the ester of the β-isomer was collected in the lower layer and washed with water to neutral reaction. The yield was about 70% (theory). The ester of the α-isomer b_{10} 64-65°, b_{760} 128-9.5°, d_4^{15} 1.0803, n_D^{15} 1.4019, M. R. 31.82; the β-isomer b_{10} 85-90°, b_{760} 151-5°, d_4^{15} 1.1101, n_D^{15} 1.4297, M. R. 31.69. The splitting off of HCl from the esters was accomplished by boiling with 1.5 parts (by wt.) of quinoline in the presence of a small amt. of hydroquinone (depolymerization agent). The yield for the α-isomer was 58% and for the β-isomer 87.4%, and the reaction proceeded twice as fast in the latter case as in the former. The product had all the properties of the Me ester of metacrylic acid. The splitting off of HCl from α- and β-chloroisoölleic acids was accomplished by boiling with 2% of activated vegetable charcoal in the presence of a small amt. of hydroquinone. The product of the reaction was distd. at 165° and had all the properties of metacrylic acid, which was esterified with MeOH. Seven references. A. A. Podgorny

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The addition of hydrogen to acetylene derivatives. XXX. Catalytic hydrogenation of symmetrical dimethyldiisopropylbutynediol. Yu. S. Zal'kind and A. I. Nogaideli. *J. Gen. Chem.* (U. S. S. R.) 8, 1382-4 (1938); cf. *C. A.* 32, 2080.—To $(iCMgBr)_2$, prepd. from 137 g. iBrBr and 32 g. Mg in 200 ml. Et_2O, is added dropwise 121 g. $Me_2CHCOMe$ at 0° in 3 hrs., the reaction mixt. allowed to stand 12 hrs., decompd. with dil. AcOH, extd. with Et_2O and the residue from Et_2O is fractionated to give $\{iCC(OH)(CHMe_2)Me\}_2$ (I), b_4 120-2°, m. 78-80° (ligroin). I in alc. contg. colloid Pd is hydrogenated to *s dimethyldiisopropylbutanediol*, b_{-4} 110-21°, d_4^{20} 0.9344, n_D^{20} 1.4665. I is hydrogenated 3 times slower than dimethyldiethylbutynediol but considerably faster than tetraethylbutynediol. Chas. Blanc

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Action of bromine upon a glycol of the ethylene series

Vu. S. Zal'kind and A. I. Nogaideli. *J. Gen. Chem.* (U. S. S. R.) 8, 1816-22(1938); cf. Zal'kind and Teterin, *C. A.* 26, 2977.—[PhCH(OH)CH:]$_2$ (I) (m. 152°) in $CHCl_3$, on treatment with Br at 0°, gives *2,5-diphenyl-3,4-dibromotetrahydrofuran* (II) and *2,5-diphenyl-3-bromo-2,5-dihydrofuran* (III). The reaction at −15° gives only II isolated by direct vacuum distn. of the solvent at room temp. It gradually, though incompletely, loses HBr to form III. Neither bromide gives CH_4 with EtMgBr. I adds I even more sluggishly to form the *monoiodide*. The reaction with [iso-PrMeC(OH)C:]$_2$ gives *2,5-dimethyl-2,5-diisopropyl-3-bromo-2,5-dihydrofuran*, d_4^{20} 1.2942. A mixt. (9 g.) of stereoisomeric dimethylbutenediols (3-hexene-2,5-diols) (IV), b_{12} 104-11° and 12.4 g. Br in 13.8 ml. $CHCl_3$ gave 0.7 g. *3,4-dibromo-2,5-hexanediol*, m. 138.5-40°, and 14.5 g. (69%) *2,5-dimethyl-3,4-dibromotetrahydrofuran*. When vacuum distd., it cleaves HBr to give *2,5-dimethyl-3-bromo-2,5-dihydrofuran*, b_6 48-51°. Ac ester of IV, prepd. by hydrogenation of hexynediol acetate (cf. Z. and Bezsonova, *C. A.* 16, 2327), gave 2 fractions, b_6 86-7° and 94-6°. The fraction, b_6 86-7°, adds 2 H atoms in the presence of the Pd catalyst to form 2,5-hexanediol, b_6 93-4°, d_4^{20} 0.9983, n_D^{20} 1.4232, M. R.$_D$ 51.68. The fraction, b_6 86-7°, reacts with Br to give the monobromoglycol, *MeCH(OAc)CH:CBrCHOAc*, b_6 92-6°.

Chas. Blanc

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Structure of several polychloro derivatives of biphenyl. Yu. S. Zal'kind and M. V. Belikova. *J. Gen. Chem.* (U. S. S. R.) 8, 1918-21 (in French, 1921) (1938).—$KMnO_4$, CrO_3 and HNO_3 (d. 1.2) have very little or no oxidizing effect on polychlorobiphenyls. Pentachlorobiphenyl (I), bp 195-230°, d^{20} 1.5, when oxidized with HNO_3 (d. 1.4) for 100 hrs. gives a mixt. of 3,4-$Cl_2C_6H_3CO_2H$ (II), m. 204-5°, and 2,4,5-$Cl_3C_6H_2CO_2H$ (III), m. 150-60.5°. Heptachlorobiphenyl (IV), bp 240-280°, d^{20} 1.64, when oxidized gives a small amt. of III and 2,3,4,5-$Cl_4C_6HCO_2H$ (V), m. 190.5-1.5°. To confirm its structure V was prepd. by heating o-$(CO_2H)_2C_6Cl_4$ in AcOH to 300° for 7 hrs. I is therefore the 2,4,5,3',4'-compd. and IV is 2,3,4,5,2',4',5'-heptachlorobiphenyl. The benzoic acids were isolated as the amides: amide of II, m. 120-30°; III, m. 167-7.5°; V, m. 200-2°. In Ph, the Cl evidently has a greater orientating influence than does Ph.

John Livak

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Some derivatives of cyclopentane. Yu. S. Zal'kind and I. F. Markov. *J. Applied Chem.* (U. S. S. R.) 11, 818-22 (in French 822) (1938); cf. *C. A.* 31, 3875[9].—The chlorination of cyclopentane at room temp. yielded 75.3-79.7% (on reacted hydrocarbon) of cyclopentyl chloride which easily split out HCl when passed over activated C with steam at 350-70° to give 88% (on reacted chloride) of cyclopentene. Treatment of the latter with 80% soln. at 22-38° yielded 81.2-82.2% cyclopentanol. Boiling the mixt. of adipic acid with a double amt. of cyclopentanol in the presence of 1% by wt. (of the adipic acid used) of concd. H_2SO_4 under a reflux condenser for 4 hrs. yielded 89.9% of dicyclopentyl adipate, m. 31-3°, b$_{10}$ 252-54°. Similar treatment of cyclopentanol with glutaric acid yielded 99.4% of dicyclopentyl glutarate, b$_{10}$ 218-20°, d_4^{20} 1.0392. Similar treatment of cyclohexanol with glutaric acid yielded about 100% dicyclohexyl glutarate, b$_{10}$ 236-8°, d_4^{20} 1.0415. Both esters had better plastic properties on nitrocellulose than dicyclohexyl adipate or tritolyl phosphate. On benzylcellulose both esters are about equal in their plastic properties to dicyclohexyl adipate and higher than tritolyl phosphate. Fifteen references.

A. A. Podgorny

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Determination of pinene in turpentine. Vu. S. Zal'kind, E. N. Rostovskii and A. G. Bulavskii. *Plasticheskie Massy, Sbornik Statei* 1939, 255-60; *Khim. Referat. Zhur.* 1940, No. 6, 74.—The relation between the amt. of turpentine distd. off below a certain temp. and the content of pinene in it was investigated. Artificial mixts. were prepd. for this purpose from pure pinene and from the residual fractions of turpentine (freed from pinene). The compns. of the mixts. obtained were similar to those of natural turpentines. Both the mixts. and the natural turpentines (in which the contents of pinene were detd. according to the method of Darmois) were distd. in an Engler-Ubbelohde app. The fractions obtained from the 1st distn. were again rectified and the fractions possessing identical coeffs. of rotation of polarized light were combined. The following physical consts. were detd. in the process of the investigations: the rotatory power for the spectrum lines 650, 578, 546 and 480 mμ, the coeff. of rotatory dispersion, b. p., α, d, and temps. of soln. of the single fractions in AcO. On the basis of the data obtained curves were constructed and a diagram was made which can be used for a rapid (approx.) detn. of pinene in turpentine. W. R. H.

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Cd

C. J. West

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Synthesis of 2-vinylnaphthalene. Yu. S. Zal'kind and I. A. Arbuzova. *Plasticheskie Massy, Sbornik Statei* No. 3, 249-55(1939); *Khim. Referat. Zhur.* 1940, No. 3, 90.—2-Vinylnaphthalene (I) is synthesized by dehydrogenation of 2-ethylnaphthalene (II). II is produced according to the modified method of Marchetti, but in insignificant amts. Better results were obtained by boiling a mixt. of naphthalene, polyethylated benzene and $AlCl_3$. This produced 55% of I (calcd. from the amt. of naphthalene used) which was identified by transforming it into the picrate. II was dehydrogenated by passing the vapors of II over catalysts consisting of a mixt. of Cr_2O_3 and MgO or of Al_2O_3 and ZnO. The reaction was carried out at a reduced pressure at 600-700° in an atm. of H. The yield of I was approx. 40-2% of the wt. of II and approx. 53-5% of the wt. of the reaction product. Pure I was obtained by distn. Owing to slight polymerization only approx. 60% of the crude product was obtained. After recrystg. several times pure I was obtained and identified by transforming it into the corresponding dibromide. By polymerizing I in the presence of Bz_2O a solid, transparent, slightly yellow polymer was obtained. Polymerization of the impure product gave the same results. References and patents for producing II and I and for polymerizing I are given.

W. R. Henn

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A method for the analysis of terpenes and the composition of Kuskovsko turpentine. Yu. S. Zal'kind and A. G. Bulavskii. *J. Gen. Chem.* (U. S. S. R.) 9, 369-78 (1939).—The method of Darmois (*C. A.* 6, 2888) is discussed and shown to be valid only for mixts. of optically active terpenes. Contrary to the results of earlier investigators turpentine obtained from *Pinus sylvestris* grown in central European Russia contains small amts. of *l*-β-pinene (I). Two com. grades of turpentine, after repeated fractional distn., have an av. compn. of 64% *d*-α-pinene (II), 6% I, 14% *d*-Δ^3-carene (III) and 12% higher-boiling products. II, b_{16} 58°, d_4^{20} 0.859, n_D^{20} 1.4660, α_e/α_f 1.14, α_f/α_c 1.96 (v = 546.1 mμ, f = 578 mμ, f = 486.1 mμ, c = 656.3 mμ), temp. of soln. in Ac_2O 53.8°. I, b. 164.0-4.5°, d_4^{20} 0.8732, n_D^{20} 1.4795, $[\alpha]_D$ −19.81°, α_e/α_f 1.066, α_f/α_c 1.430. III, b_{16} 68°, d_4^{20} 0.865, n_D^{20} 1.4728, α_e/α_f 1.16, α_f/α_c 2.17, temp. of soln. in Ac_2O 39°. The presence of I is shown by oxidation with alk. $KMnO_4$ to give nopinic acid, m. 128-7°, and by conversion to borneol, m. 198-201.5°. Owing to the anomalous values obtained the dispersion coeffs. α_e/α_f and α_f/α_c possess little value for characterization of mixts. contg. I and *d*-rotatory terpenes (II, III). John Livak

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C A

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A method for determining the gas-permeability (of refractories). D. N. Polubojarinov and I. Ya. Zalking. *Ogneupory* 1939, No. 9, 611-14; *Khim. Referat. Zhur.* 1940, No. 3, 80.—Methods for detg. gas-permeability of refractories require measuring the vol. of air passing through the sample, the time and the air pressure. A simplified method is proposed which requires only the measuring of the working pressure of the air; an equation is derived for detg. the gas permeability. W. R. Henn

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The addition of hydrogen to acetylene derivatives. XXXI. Catalytic hydrogenation of acetylene γ-glycols containing cyclopentyl radicals. Yu. S. Zel'kind and I. M. Gverdtsiteli. *J. Gen. Chem.* (U. S. S. R.) 9, 855-62 (1939); cf. *C. A.* 33, 4105[1].—To $(ICMgBr)_2$, prepd. from 12 g. Mg, 55 g. EtBr and CH:CH, there is added 42 g. cyclopentanone while cooling and stirring. After 24 hrs. the mixt. is decompd. and the reaction product extd. with Et_2O. The residue from Et_2O gives bis(1-hydroxycyclopentyl)acetylene (I), m. 100.8-10.8°, in 45.2% yield. I yields acetylene on warming with 40% KOH soln. and gives a violet color with concd. H_2SO_4. It is slightly sol. in C_6H_6, rather sol. in alc. and Et_2O. The filtrate from I gives 1-ethynyl-1-hydroxycyclopentane, b. 145-50°. I in alc. contg. colloidal Pd is hydrogenated to bis(1-hydroxycyclopentyl)ethylene (II) which is obtained in 2 isomeric forms, m. 129.6-30.6° (III) and 82-3° (IV), resp. Both isomers give a bright-violet color with H_2SO_4. 1,2-Bis(1-hydroxycyclopentyl)ethane (V), m. 131.2-1.4°, is obtained from I on hydrogenation in the presence of Pt. III and IV yield the γ-oxide, m. 81-2°, on heating with a crystal of I; it is also obtained on warming III with H_2SO_4. The diacetate of I, m. 44.5-5.5°, prepd. by heating 12 g. I, 36.5 g. Ac_2O and 1 g. NaOAc, is easily sol. in alc., Et_2O, ligroin and MeOH and yields on hydrogenation in the presence of colloidal Pd the diacetate of V, b. 108-10° (partial decompn.), d^{18} 1.0004, n_D^{18} 1.47507. When the hydrogenation is stopped after the addn. of 2 atoms H, the diacetate of II is obtained, b. 120.5-9° (partial decompn.), d^{18} 1.072. To EtMgBr prepd. from 24 g. Mg and 110 g. EtBr is added in small portions 42 g. dimethylethynylcarbinol within 1.5 hrs. while cooling and stirring. The mixt. is heated for 15 min. and allowed to stand for 2-3 hrs. Then 42 g. cyclopentanone is added slowly while cooling. After 24 hrs. the mixt. is heated with moist Et_2O. The residue from Et_2O is distd. with steam, and the residue from the steam distn. is extd. with Et_2O. After evapn. of the Et_2O dimethyl(1-hydroxycyclopentylethynyl)carbinol (VI) m. 56-8°, b. 125-6°, is obtained, which is easily sol. in org. solvents. Hydrogenation of VI in the presence of colloidal Pd gives dimethyl(1-hydroxycyclopentylvinyl)carbinol, m. 89-90°. G. B.

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The synthesis of an alcohol with two conjugated triple bonds. Yu. S. Zal'kind and M. A. Afzikovich. *J. Gen. Chem.* (U. S. S. R.) 9, 901–4(1939).—On heating 10 g. 2,7-dimethyl-3,5-octadiyne-2,7-diol with 20 g. $Ca(OH)_2$ at 75–130°, it is cleaved, giving a mixt. of Me_2CO, biacetylene and traces of *5-methyl-1,7-hexadiyn-3-ol*, $Me_2C(OH)C{\equiv}CC{\equiv}CH$ (I). The same results were obtained by using K_2CO_3 or $Ba(OH)_2$. However, on heating the above reactants for only 30 min., i. e., terminating the reaction before its completion at 120–30°, 17% (theory) of I was obtained. In this case, 46.5% of glycol was recovered. I b_7 50–61°, b_{14} 72–4°, d_4^{20} 0.9395, n_D^{20} 1.4850, MR_D = 33.00. Since I is not very stable, it was extd. from the reaction mixt., after distg. off most of the biacetylene and Me_2CO, by NH_3-Ag_2O soln. and the resulting ppt. was decompd. (cold) with 20% H_2SO_4 under an Et_2O layer (several days required). The I formed immediately dissolved in the Et_2O layer, and was recovered from it in the usual manner. Biacetylene glycols with 2 conjugated triple bonds on heating with K_2CO_3, $Ca(OH)_2$ or $Ba(OH)_2$, decomp., forming 2 mols. of ketone and biacetylene or 1 mol. of ketone and biacetylene alc.

A. A. Podgorny

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Synthesis of glycols of the biacetylene series. Yu. S. Zal'kind and I. M. Gverdtsiteli. *J. Gen. Chem.* (U. S. S. R.) 9, 971-4(1939); cf. *C. A.* 31, 4283[1]. The previously described method for the synthesis of biacetylene glycols was used in the prepn. of *bis(1-hydroxycyclopentyl)-biacetylene* (I), m. 133.2-4.2°, from 1-hydroxycyclopentylacetylene. The yield was almost quant. I, hydrogenated in the presence of Pt black, added 8 H atoms, forming *1,4-di(1-hydroxycyclopentyl)butane*, m. 91.8-92.5°. In the presence of colloidal Pd, the hydrogenation after addn. of 4 H atoms proceeded very slowly. Stopping the reaction at this stage yielded, probably, *1,4-di(1-hydroxycyclopentyl)-1,3-butadiene*, m. 101-4°. The application of this reaction to the synthesis of biacetylene derivs. of OH compds., using secondary acetylene alcs., was also investigated. For this purpose, the following alcs. were prepd. by the Teterin and Ivanova method (cf. *C. A.* 14, 8303[1]) from the corresponding aldehydes, BrMgC₂CMgBr and alc.: *1-butyn-3-ol*, b. 107-9°; *1-pentyn-3-ol*, b. 124-6°, d^{20} 0.8851, n_D^{20} 1.43447, MRD 24.74; *1-hexyn-3-ol*, b. 140-1°, d^{20} 0.8704, n_D^{20} 1.43297, MRD 29.38, and *phenylethynylcarbinol*, PhCH(OH)C:CH, b_{14} 114-15°, d^{18} 1.0215, n_D^{18} 1.5201, MRD 39.815. In all cases the yield was 20-35% (theory). The condensation of these alcs. yielded, in very small amts.: *3,5-octadiene-2,7-diol*, in 2 isomeric forms, m. 107-9° and 67-9°; *4,6-decadiyne-3,8-diol*, [MeCH₂CH(OH)C:C]₂, b. 154-6°; *3,7-dodecadiyne-4,9-diol*, [MeCH₂CH₂CH(OH)C:C]₂, b. 150-62°; and *1,6-diphenyl-2,4-hexadiyne-1,6-diol*, [PhCH(OH)C:C]₂, m. 132-3°. The condensation probably proceeded through the formation of free radicals by removal of a H atom with subsequent condensation of free radicals. The formation of free radicals in the case of secondary alcs. is retarded because the pos. inductive effect of Me group, which promotes this process, is balanced by the neg. effect of OH, whereas in the case of tertiary alcs. the effect of 2 Me groups is only partially balanced by 1 OH group. This explains the low yields of biacetylene derivs. of OH compds. A. A. Podgorny

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C-1

The reaction between acetylenedimagnesium dibromide and carbonyl compounds. Yu. S. Zal'kind and S. M. Lalusov. *J. Gen. Chem.* (U.S.S.R.) 9, 1523-32(1939). —The influence of the structure of CO compds. on the condensation of aldehydes and ketones with $(BrMgO)_2$ (I) in the synthesis of acetylene γ-glycols by the Iotzich method is studied. To this end, I, prepd. from 2 g. Mg, 12 g. EtBr and 100 ml. ether, is treated at 0° with a definite quantity of an aldehyde and ketone for measured periods of time and the reaction mixt. is decompd. with H_2O contg. H_2SO_4. The vol. of liberated C_2H_2 gives the amt. of unaltered I and indirectly the C_2H_2 used in the reaction. The exact content of I in the reagent was detd. by expelling the ether and decompg. the residue with H_2O. It was 915 ± 10 ml. C_2H_2, corresponding to 98.06% I. This figure was used in calcg. the results of the condensation reactions. Furthermore, since some C_2H_2 is dissolved by ether in the I reagent, its mean content was detd. by decompg. the reagent with water without the preliminary removal of the ether and heating the mixt. on a water bath. A vol. of 1249 ml. C_2H_2 was collected or 331 ml. in excess of the amt. liberated in the decompn. of I reagent with the previous expulsion of the ether. This C_2H_2 amt. was deducted from the C_2H_2 formed in the decompn. of the condensation reaction mixt. In the condensation with Me_2CO, MeCOEt, MeCOPr, Et_2CO, MeCOPh and Ph_2CO the reaction proceeds rapidly in the first 15 min. with the decompn. of 59.63% I and reaches a max. of 57.8% in 1 hr. This limit is not exceeded by longer duration of the reaction (4 hrs.) or by the use of a large excess of ketones (10 times). In the presence of I excess the condensation proceeds nearly quantitatively. The results show that the reaction is not reversible, hence the incomplete condensation is not caused by a reaction equil. in conformity with the law of mass action. It is probable that the reaction stops because of the occlusion of I by the insol. Mg complex formed in the reaction. The reaction proceeds in 2 stages: $Me_2CO + I \rightarrow Me_2C(OMgBr)C{\cdot}CMgBr$; $Me_2C(OMgBr)C{\cdot}CMgBr + Me_2CO \rightarrow [Me_2C(OMgBr)C]_2$. This is confirmed by the formation of a glycol (tetramethyl-2-butyne-1,4-diol) and alc. $(Me_2C(OH)C{\cdot}CH)$. In general, the velocity of condensation decreases with increasing mol. wt. of ketones. Aldehydes (AcH, EtCHO, PrCHO and BzH) react with I more rapidly to a max. of 70.16% decompn. (AcH).

Chas. Blanc

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CA

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Preparation of substituted diphenylbiacetylenes. Yu. S. Zal'kind and B. M. Fundyler. *J. Gen. Chem.* (U. S. S. R.) 9, 1723-6(1939).—In a previous paper the authors described the synthesis of PhC:CC:CPh from PhC:CH and NH_4-CuCl. Similarly tertiary alcs. with acetylenic radicals form corresponding diacetylenic glycols, a reaction which is unsuccessful with secondary acetylenic alcs. (low yields). p-Methyl-, p-chloro-, p-bromo- and p-nitrophenylacetylenes were subjected to this reaction to check on the electronic theory of Ingold and Robinson. The following yields and time of complex formation were noticed: p-tolyl 90%, immediate; Ph 75%, 25-30 min.; p-ClC_6H_4 50%, 30-5 min.; p-BrC_6H_4 45%, 45-50 min.; p-$O_2NC_6H_4$ 28%, 90-120 min. *tert*-BuC:CH yielded di-*tert*-butylbiacetylene. p-Tolylacetylene was prepd. according to Kunckell and Goetsch (*Ber.* 33, 2356 (1900)) by the action of Na on p-MeC_6H_4CCl:CHCl, obtained from $MeC_6H_4COCH_2Cl$ and PCl_5. p-Tolylacetylene (2 g.) was added dropwise to a soln. of 10 g. of CuCl, 16 g. of NH_3 and 0.15 cc. of concd. HCl in 48 cc. of H_2O at 55-65°. The formation of a solid complex was immediate. Heating lasted for 3 hrs. A current of air was continuously passed through the mixt. When treated with ether the complex decompd. A 90% yield of di-p-tolylbiacetylene, m. 183-3.2°, was obtained. A mixt. of 85 g. $ClC_6H_4CCl_2Me$, b_{10-15} 110-18°, and 8 g. ClC_6H_4CCl:CH_2, b_{10-15} 104°, was prepd. from 94 g. of p-chloroacetophenone and PCl_5. These chlorides were boiled 3 hrs. with 600 g. of 25% KOH in alc. After allowing the mixt. to stand for 24 hrs. the product was treated with H_2O, extd. with ether, dried and vacuum-distd., $b_{0.15}$ 85-95°, m. 41-3°. Di-p-chlorophenylbiacetylene was obtained in 50% yield, m. 258°. Di-p-bromophenylbiacetylene, m. 263-4°, was prepd. similarly to the Cl deriv. p-Nitrophenylacetylene was prepd. according to Drewsen, *Ann.* 212, 154. It was condensed as in previous expts., yielding di-p-nitrophenylbiacetylene, m. 285-6°.

D. Aelony

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Preparation of cyclohexanol from cyclohexene. Yu. N. Zel'kind and I. P. Markov. *J. Applied Chem.* (U. S. S. R.) 12, 437-9 (in French, 439) (1939).—Mixing 1 mol. of cyclohexene with 1-1.5 mols. of 70-85% H_2SO_4 at 54-6° for 50-60 min., subsequent dilu. of the reaction mixt. with 5 vols. of water, steam distg., salting out with NaCl and drying with K_2CO_3 yielded, after fractionation, 50-54% of cyclohexanol. The cyclohexene obtained from petroleum yielded less cyclohexanol, probably because of impurities. A Ag_2SO_4 catalyst slightly increased the yield and decreased the tar formation. A $K_4Fe(CN)_6.3H_2O$ catalyst had no effect.

A. A. Podgorny

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3RD AND 4TH ORDERS

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The chlorination of carbazole. Yu. S. Zal'kind and M. E. Komarenko. *J. Applied Chem.* (U. S. S. R.) 12, 1134-6(in French, 1136)(1939).—Dry Cl was passed through 1 g. carbazole in 20 cc. of CCl_4, yielding 30-2% (by wt. of carbazole) white crystals of tetrachlorocarbazole, m. 223-4°, sol. in C_6H_6, PhMe, $CHCl_3$, Et_2O, Me_2CO, CCl_4 and alc. The chlorination of the carbazole (0.5 g.) or tetrachlorocarbazole in CCl_4 in the presence of some cryst. I yielded white crystals of octachlorocarbazole, m. 276-7°, sol. in C_6H_6 and PhMe, slightly in $CHCl_3$ and ether and insol. in cold alc. and Me_2CO.

A. A. Podgorny

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Investigation of the Malter effect. M. S. Kosman, Yu. E. Zal'kind, and M. E. Zal'kind. *Uchenye Zapiski Leningrad. Gosudarst. Univ., Ser. Fiz. Nauk* **1940**, 40-5 (in Russian); cf. Malter, *Phys. Rev.* **50**, 48 (1936).—(1) The abnormally high secondary electron-emission current from Al_2O_3 on Al and the persistence of the effect after the end of the primary bombardment were further confirmed on 4 different dielectrics, Al_2O_3, mica 3 μ and 10 μ thick, and MgO, all on a grounded Ni support. The basic condition for the after-effect appears to be suppression of the elec. surface cond.; with well-outgassed mica, the Malter current intensity i drops only by 2-3% in 30 min., but the drop is much faster with insufficient outgassing. Especially with powders (MgO), because of inhomogeneity of the surface, sudden changes in i occur to the extent of 100% in either direction; in this case, i may persist for up to 4-5 hrs. with only slight fluctuations and then suddenly disappear. (2) At a given primary current intensity I, the Malter current i increases steadily with the collector voltage V; this holds also for the after-effect ($I = 0$), which indicates that at least part of the electrons are emitted by the dielectric. There are evidently 2 processes present, emission from the dielectric without replacement by electrons from the metal support, resulting in raising the potential of the surface, and emission with replacement, the second process detg. the stationary state. Indication for the first process is seen in the observation that the Malter current "revives" spontaneously, that is without primary bombardment, when the collector potential is again applied after having been lowered to $V = 0$; discrete centers in which the pos. potential is coned., assumed to be present on the dielectric, evidently persist at $V = 0$ and on its renewed application give rise to emission by the first mechanism until the surface potential reaches V. Primary bombardment at $V = 0$ liquidates the centers and suppresses subsequent spontaneous revival. (3) Thoroughly outgassed films show no dependence of i on I. With MgO powders with outspoken elec. cond., i increases with I in nearly the same way for various values of V; the effect of cond. tends to diminish with rising I, hence i tends to satn. (4) Electron velocity distribution curves at high i show most of the electrons to have but low velocity, only a negligible fraction having velocities corresponding to the potential drop across the dielec. layer. This indicates that the majority of electrons are emitted from the shallowest depths of the film; the alternative interpretation, namely that electrons are emitted from the metal support but lose energy on traversing the film, is contradicted by the curve of angular distribution showing the majority of electrons to be emitted perpendicular to the surface and absence of any noticeable scattering such as would result from loss of energy through collisions. At low i, there is a substantial no. of electrons having full velocity; this indicates that in this case most electrons are emitted from the metal support. N. Thon

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A. E. Favorskiĭ and his work. Yu. S. Zal'kind. Uspekhi Khim. 9, 152-64(1940). Reminiscences of the first years of the scientific and pedagogical work of A. E. Favorskiĭ. V. E. Tishchenko. Ibid. 165-80.
P. H. Rathmann

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PROCESSES AND PROPERTIES INDEX

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C4

The addition of bromine and chlorine to 2,3-diphenylbutadiene. Yu. S. Zal'kind and P. Musunov. J. Gen. Chem. (U. S. S. R.) 10, 817-20(1940). Br acts on CH_2:CPhCPh:CH_2 in $CHCl_3$ or petr. ether by 1,4-addn. to give 1,4-dibromo-2,3-diphenyl-2-butene, m. 149-50°, which is oxidized by O_2 to $BrCH_2COPh$. Similarly, Cl forms *1,4-dichloro-2,3-diphenyl-2-butene*, m. 143-4°.
H. M. Leicester

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CA

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The addition of hydrogen to acetylene compounds. XXXII. Catalytic hydrogenation of an alcohol with a double and a triple bond. Yu. S. Zal'kind and N. D. Khudekova. *J. Gen. Chem.* (U. S. S. R.) 10, 521-6 (1940); cf. *C. A.* 34, 3874.—CH_3:CHC:CH, EtBr and Mg with MeEtCO give 77.5-88% MeEtC(OH)C:CCH:CH_3, b_{20} 75-6°, b_{18} 81-2°. When this is hydrogenated over Pt black it easily adds 4 H atoms, then in about double the time it adds 2 more. A very slow reaction with H follows, probably due to reduction of the OH group. When colloidal Pd deposited on starch is used, the addn. of the last 2 H atoms takes 3-4 times as long as addn. of the first 4, and there is no further reaction. Addn. of the first 2 H atoms goes almost entirely on the triple bond, giving a compd. which reacts with maleic anhydride to form crystals m. 136-7°. The next 2 H atoms add to any of the available double bonds, giving a mixt. of unsatd. alcs. When 6 atoms of H have been added, the product is MeEtBuCOH

H. M. Leicester

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Fixation of carboxylic acids with acetylenic γ-glycols. Vu. S. Zal'kind and V. I. Baranov. *J. Gen. Chem.* (U. S. S. R.) 10, 1432-4(1940).- $Me_2C(OH)C\equiv CC(OH)Me_2$ was added in 2 portions (15 and 10 g.) to a mixt. of 35 cc. AcOH, 15 cc. BF_3 soln. in AcOH and 1 g. $Hg(OAc)_2$ in 15 cc. of AcOH, while heating at 100-15° for 15 hrs. The following products were identified: 2,2,5,5-tetramethyltetrahydro-3-ketofuran, $b_{0.5}$ 40.5-2°, and 2,2,5,5-tetramethyl-2,5-dihydro-3-hydroxyfuran acetate, $b_{0.5}$ 88-90°, m. 30.5-1°. A. A. Podgorny

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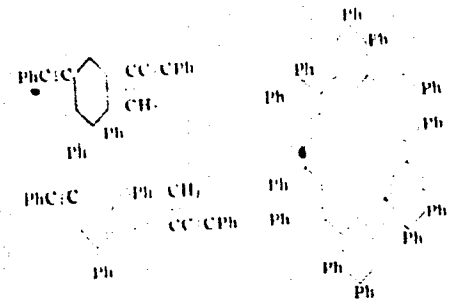
(A)

Dehydration of alcohols containing two triple bonds and the products of this reaction. Yu. S. Zal'kind and E. F. Zeberg. *J. Gen. Chem.* (U. S. S. R.) 10, 1798-1812 (1940).—Prepn. of hydrocarbons of type R_4C $C(C:CR)_2$ was attempted by dehydration of the corresponding diethynyl alcs. $(PhC:C)_2C(OH)Me$ (I), from PhC:CMgBr and EtOAc, was dehydrated by H_2SO_4, H_3PO_4, $KHSO_4$, $(CO_2H)_2$, p-$MeC_6H_4SO_3H$, yielding in all cases only the polymer of the desired hydrocarbon: *unsym-bis(phenylethynyl)ethylene* (II). All work was done in an inert atm. due to sensitivity of the products. The latter were brownish powders which were fractionated by benzene-EtOH pptns. into a series from the dimer to the octamer of II. The dimer and trimer were unsatd. and tended to polymerize further on standing, while the higher polymers were stable. The following formulas are proposed for the lower and higher polymers:



Hexylbis(phenylethynyl)carbinol (III), from EtMgBr, PhC:CH and Et heptanoate, was prepd. in order to study the effect of a larger group R. However, dehydration of III by 60% H_3PO_4 also yielded polymeric material only, as an alc.-insol. brown solid, apparently a tetramer of the desired hydrocarbon. III m. 65-5.5° (from petr. ether).

G. M. Kosolapoff

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The addition of hydrogen to acetylene compounds. XXXIII. Synthesis and catalytic hydrogenation of hydroxy acids of the acetylene series. Yu. S. Zal'kind and B. I. Mikhant'ev. *J. Gen. Chem.* (U. S. S. R.) 11, 92-8 (1941); cf. *C. A.* 34, 7847.—When CO_2 is passed through Me_2CO and the mixed vapors are passed into BrMgC:C-MgBr (I) at −8°, 0.75% *1,1-dimethyl-2-butyn-1-ol-4-carboxylic acid* (II), decomp., on vacuum distn., is obtained. When $Me_2C(OMgBr)C{:}CMgBr$ (III) is treated with CO_2 at 8°, the yield of II is 1.25%. This can be raised somewhat if the CO_2 is passed into the mixt. for 5 days. Treatment of III with $ClCO_2Me$ gives 1.5% II. The Ag, Cu, Ba and Ca salts of II are insol. The Ag salt is explosive. The *amide* m. 72-3°. In similar reactions I, Ph_2CO and CO_2 give 0.7% *1,1-diphenyl-2-butyn-1-ol-4-carboxylic acid* (IV), m. 78-80°, decomps. 127°. $Ph_2C(OMgBr)C{:}CMgBr$ (V) and CO_2 give 3.6% IV, and V with $ClCO_2Me$ gives 0.44% IV. The Ag, Cu, Ca, Ba, Sn and Pb salts of IV are insol.; the *amide* m. 96-7°. Hydrogenation of II, IV and their amides over Pd and Pt adds 4 H atoms. The rate of addn. over Pt is regular, but over Pd the last 2 H atoms add more slowly. This effect is not as marked as in the acetylene γ-glycols however.

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3RD AND 4TH ORDERS

Ca

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Addition of hydrogen to acetylenic derivatives. AAA-III (?). Catalytic hydrogenation of phenylated butynediols in the presence of colloidal palladium. Vu. S. Zal'kind and A. P. Ivanov. *J. Gen. Chem.* (U. S. S. R.) 11, 1667-10 (1941); cf. C. A. 35, 6569[2].—The authors made a comparative study of the hydrogenation of 10 acetylenic glycols in the presence of colloidal Pd under controlled conditions. BrMgC CMgBr (from 30 g. Mg) was treated, with cooling and stirring, with 60 g. MeCOPh and 18 g. abs. EtOH in dry Et_2O, let stand 24 hrs., treated with moist Et_2O, the aq. layer treated with weak H_2SO_4 and Et_2O; both Et_2O exts. were dried and the Et_2O distd.; the 1st ext. yielded a mixt. of *sym-dimethyldiphenylbutynediol* and *2-phenyl-3-butyn-2-ol* (I); the latter was dissolved in warm petr. ether, the solvent distd. and the residue steam-distd., the distillate satd. with K_2CO_3 and extd. with Et_2O. The 2nd Et_2O ext. treated as above also yielded I, giving a total of 21.5 g. I (29.4%), m. 48.5-9° (from petr. ether). EtMgBr (from 14.4 g. Mg) in ether was treated with 24 g. dimethylethynylcarbinol in Et_2O with cooling and stirring, followed by 24 g. BzH in ether; after 2 days the mixt. was treated with moist Et_2O and ice water, and the aq. layer treated with dil. H_2SO_4, and extd. with Et_2O. On distn. of the Et_2O both exts. gave crystals of *4-methyl-1-phenyl-2-pentyne-1,4-diol*, (II), which was recrystd. from benzene and EtOH; 20 g. (46.4%), m. 73.5-4°. EtMgBr (from 3.6 g. Mg), treated with 9.0 g. phenylethynylcarbinol with cooling and stirring, warmed for 0.5 hr. on a water bath, then treated with 9.4 g. Ph_2CO in ether and let stand for 3 days, heated for 0.5 hr. on a steam bath and worked up as above, gave 11 g. (67.8%) *1,1,4-triphenyl-2-butyne-1,4-diol* (III). III was also prepd. from EtMgBr (from 2.5 g. Mg), 10.4 g. diphenylethynylcarbinol and 5 g. BzH in 64.2% yield. III, m. 140-0.5°. *2-Methyl-5-phenyl-3-hexyne-2,5-diol* (IV), prepd. as described above from 14.4 g. Mg, 66 g. EtBr, 24 g. dimethylethynylcarbinol and 27 g. MeCOPh, in 76.2% yield (35 g.), m. 81.5-2°. *1,1,4-Triphenyl-2-pentyne-1,4-diol* (V), prepd. as described above from 4.8 g. Mg, 22 g. EtBr, 14.6 g. I, and 15 g. Ph_2CO, in 34.6% yield (9.4 g.), m. 123.5-4°. *3-Methyl-1-(9-hydroxy-9-fluorenyl)-1-butyn-3-ol* (VI), prepd. from 37 g. fluorenone, 18 g. Mg, 88 g. EtBr and 34 g. dimethylethynylcarbinol as described above, was obtained in 62.7% yield (34 g.), m. 163-3.5° (from EtOH). The 5 glycols and 5 previously known ones: diphenyl-, tetramethyl-, *unsym*-dimethyldiphenyl-, *sym*-dimethyldiphenyl- and tetraphenylbutynediol, were hydrogenated alike, by using 0.01 g. mol. of the glycol in 50 cc. EtOAc and 2 mg. colloidal Pd. All but V and tetraphenylbutynediol were run at 19° and 743 mm., the remaining 2 were run at 17.5° and 743 mm., and 20° and 758.5 mm., resp. In addn., expts. were also run with 4 mg. Pd catalyst, and all new glycols (II-VI) were also run with 6 mg. Pd. The reaction consts. (× 10^3) for 3 mg. Pd were found to be: diphenylbutynediol 9.5, II 6.16, III 3.3, tetramethyl-

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butynediol 0.6, IV 3.6, *anti*-dimethyldiphenylbutynediol 3.4, *syn*-dimethyldiphenylbutynediol 1.37, V 0.80, tetraphenylbutynediol 0.43, VI 4.09. For 4 mg. Pd the values were. 13.2, 11.2, 7.09, 15.1, 10.4, 8.5, ——, 2.1, 0.65 and 11.3, resp. Hydrogenation with 6 mg. Pd was run mainly to observe any rapid change of rate of hydrogenation after addn. of the 1st 2 H atoms; in all cases a definite decrease of hydrogenation rate was observed after formation of the ethylene glycol-type structure. Although the reaction, in general, is not strictly a 1st-order reaction, and K varies in the course of its progress, the av. values of K (given above) were calcd. because they are convenient measuring comparison standards. The products of hydrogenation of II-VI were isolated as follows: the Pd was filtered off, the H_2OAc distd. *in vacuo*, the residue dissolved in H_2O, dried, the H_2O distd. off and the residue distd. if possible. II yielded *4-methyl-1-phenyl-2-pentene-1,4-diol*, $b_{0.5}$ 154-9°; III yielded *1,1,4-triphenyl-2-butene-1,4-diol*, a viscous liquid which could not be distd. without decompn. IV yielded *2-methyl-3-phenyl-3-hexene-2,5-diol*, b_5 175-8°; V yielded *1,1,4-triphenyl-2-pentene-1,4-diol*, m. 75-6° (from benzene); VI gave *3-methyl-1-(9-hydroxy-9-fluorenyl)-1-buten-3-ol*, m. 152-3° (from C_6H_6). In general, discondary and secondary-tertiary glycols hydrogenate more readily than do the ditertiary glycols of comparable mol. wt. Substitution of Ph groups for Me groups leads to a slower hydrogenation rate. Closure of 2 Ph groups into a fluorene nucleus causes a great increase in hydrogenation rate, showing the effect of steric structure of the mol.

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Preparation of dibasic acids from petroleum fractions

Yu. S. Zal'kind and A. Khutkivadze. *J. Applied Chem.* (U.S.S.R.) 14, 405-9(1941); Translated in *Foreign Petroleum Tech.* 9, 444-52(1941).—The kerosene fraction, b. 210-15°, gave a higher yield of dibasic acids (12.20%) than other fractions on treating it with 6-7 parts of HNO_3 (d. 1.4), added in batches while heating the mixt. for 30-35 hrs. The yield was raised to 14.93% by using V_2O_5 catalyst. Adipic, glutaric and succinic acids were obtained. Decalin, when oxidized with HNO_3, forms glutaric and oxalic acids, and in the presence of other petroleum hydrocarbons, adipic acid is probably formed also. Butyldecalin on being oxidized with HNO_3 yielded adipic and glutaric acids. 8 references. A. A. Boehtlingk

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Ad

Addition of H to acetylenic derivatives in the presence of Pd or Pt. XXXVII. Catalytic hydrogenation of acetic ester of acetylenic erythritol. Yu. S. Zal'kind and A. Kakhniashvili. *J. Gen. Chem.* (U. S. S. R.) 12, 649-52 (1942)(English summary); cf. *C. A.* 36, 3790⁹.—BrMgC:CMgBr (from 24 g. Mg) was treated with 80 g. benzoin in 200 cc. dry Et_2O, let stand 3 days, heated to 60° for 3 hrs. and treated with dil. H_2SO_4 to yield *1,2,5,6-tetraphenyl-3-hexyne-1,2,5,6-tetrol* (64%), m. 218-15° (from EtOH); *tetraacetate* (by heating with Ac_2O and NaOAc in the presence of pyridine), m. 185.5-7° (from MePh). The latter hydrogenated in the presence of colloidal Pd at 17° in EtOAc gave the corresponding *ethylene compd.*, $C_{34}H_{30}O_8$, m. 181.5-3.5° (from MePh). After this addn. of 2 H the reaction slows down greatly with Pd. However, hydrogenation over Pt black in EtOAc yielded *1,2,5,6-tetraphenyl-1,2,5,6-hexanetetrol tetraacetate*, m. 187-8.5° (from MePh). G. M. K.

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Plasticizers from naphthenic acid esters. Yu. S. Zal'kind, V. P. Goguadze, and N. K. Iremadze (Tbilisi Chem. Inst.). *Bull. Acad. Sci. Georgian S.S.S.R.* 4, 087-92 (1943) (in Georgian and Russian). —The acids were extd. from two kerosene (a) 100° and 150-100°) and two oil (85-100° and 150-210°) fractions of Baku petroleum distd. under 3-6 mm. and esterified for 24 hrs. on a water bath with 1% *p*-toluenesulfonic acid as catalyst with EtOH (4-fold excess) and with glycerol (2-fold excess), in the latter case dild. with gasoline which is subsequently distd. off. From the kerosene acids one obtains Et esters m. −75, −73; glycerol esters m. −75, −21; from the oil acids, Et esters m. −66, −68; glycerol esters m. −50, −52°. By volatility tests (loss of wt. in air after 15 days), Et esters of the lower fractions are too volatile for use as plasticizers and not volatile enough for use as solvents. Esters of the higher fractions, particularly glycerides, have the right low volatility. No turbidity or pptn. occurs on mixing with cellulose ester solns. A cellulosic lacquer prepd. with naphthenic acid glyceride as plasticizer proved satisfactory. N. Thon

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The addition of hydrogen to acetylenic derivatives. XXXVI. Catalytic hydrogenation of alcohols with vinylacetylene radicals. Yu. S. Zal'kind and T. A. Boroda. *J. Gen. Chem.* (U.S.S.R.) 15, 90-108(1945)(English summary); cf. *C.A.* 38, 1470[6].—When a mixt. of Et-MgBr and CH_2:CHC:CH is treated with cyclopentanone, it gives 38-40% 1-(3-buten-1-ynyl)cyclopentanol (I), b_6 83-4°, b_7 85-6°, d_4^{20} 0.9715, n_D^{20} 1.50191, MR_D calcd. 46.62, found, 41.33. A similar reaction with Et_2CO gives 55% diethyl(3-buten-1-ynyl)carbinol (II), $b_{4.5}$ 58-8.5°, $b_{6.4-6}$ 63.5-4°, d_4^{20} 0.8983, d_4^{20} 0.8875, n_D^{20} 1.4770, n_D^{20} 1.4800, MR_D calcd. 42.82, found, 43.83. With Pr_2CO, the yield is 57.2% dipropyl(3-buten-1-ynyl)carbinol (III), $b_{4.4-6}$ 92-4°, d_4^{20} 0.8736, n_D^{20} 1.4743, MR_D found 53.43. When hydrogenated over Pt black, all 3 compds. add 6 H atoms to give resp. 1-butylcyclopentanol, b_{10} 194-4.5°, $d_4^{20.1}$ 0.8684, $n_D^{20.1}$ 1.4530, MR_D calcd. 42.90, found, 43.08; diethylbutylcarbinol, $b_{10.4}$ 178-6°, d_4^{20} 0.8312, n_D^{20} 1.4352, MR_D calcd. 45.20, found, 45.22; and dipropylbutylcarbinol, b_{10} 98-9°, d_4^{20} 0.8314, n_D^{20} 1.4399, MR_D found 54.51. Hydrogenation is fastest with II and slowest with III. When the compds. are hydrogenated over Pd, the fastest reaction is with I and the slowest with II. In these cases, only 4 H atoms add easily, and with II and III, the last 2 H atoms cannot be added. Although there is no break in the hydrogenation curves when 2 H atoms are added over Pd, if hydrogenation is interrupted at this point the corresponding diene is obtained. Under these conditions, I gives 1-(1-hydroxycyclopentyl)-1,3-butadiene, b_6 73-5°, d_4^{20} 0.9622, n_D^{20} 1.50499, MR calcd. 41.89, found 42.36 (*maleic anhydride addn. compd.* m. 127°), II gives 5-ethyl-1,3-heptadien-5-ol, b_6 59-61°, d_4^{20} 0.8684, n_D^{20} 1.4647, MR calcd. 44.35, found 44.55 (*maleic anhydride addn. compd.* m. 132°), and III gives 5-propyl-1,3-octadien-5-ol, b_6 78-9°, d_4^{20} 0.8634, n_D^{20} 1.4648, MR_D calcd. 53.59, found 53.83 (*maleic anhydride addn. compd.* m. 138°). Addn. of 2 more H atoms to these compds. occurs almost exclusively on the individual double bonds and not at the ends of the conjugated chain. The failure to add the last 2 H atoms is not due to poisoning of the Pd catalyst, but to the structure of the ethylenic compds.

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Azo derivatives of the phenanthrene series. Yu. S. Zal'kind and Z. I. Khoffen (Leningrad Chem. Tech. Inst.). *J. Gen. Chem.* (U.S.S.R.) 15, 368-74(1945)(*English summary*).—Eight azo dyes based on phenanthrene were prepd. and their spectra are presented. The 3-phenanthryl nucleus causes but slight deepening of color as compared with C_6H_5, while the 2-phenanthryl radical causes substantial color deepening. Phenanthrene (100 g.) was treated at 143° over 20 min. with 54 g. concd. H_2SO_4, after which the mixt. was heated for 20 min. longer; after pouring into 1 l. 20% NaCl and neutralization with NaOH the mixt. was allowed to stand for 24 hrs., when the ppt. was filtered off, recrystd. from 1.5 l. water, dissolved in 2 l. H_2O, and, after addn. of a little HCl, was treated with a soln. of 18 g. $BaCl_2$. The pptd. Ba salt was thoroughly washed with hot water and the residue collected as Ba 2-phenanthrenesulfonate (15.5%). The filtrates on concn. and treatment with 5 g. BaCl gave 10-11 g. more of the above Ba salt contaminated with some of the 3-isomer; repetition of the procedure yields an unspecified amt. of the pure 2-isomer. All filtrates were treated with H_2SO_4, neutralized, after filtration, with KOH and treated with KCl while hot to yield 32 g. of the K 3-phenanthrenesulfonate; addnl. amts. of this acid were obtained as the Ba salt from the filtrate by addn. of $BaCl_2$; the total yield of the 3-isomer was 31.6%. Fusion with an equal mixt. of KOH-NaOH at 250-300° gave 87% 3-phenanthrol (from the 3-sulfonic acid, as K salt), m. 120-1°. Fusion of Ba 2-phenanthrenesulfonate with 3 parts of KOH at 310-20° gave 81.5% 2-phenanthrol, m. 168.9° (from ligroin). Coupling of these phenols with the appropriate diazonium soln. in the presence of NaOH gave the following dyes: *4-phenylazo-2-phenanthrol*, m. 145-6° (from EtOH), deep red (89.1%); *1-phenylazo-2-phenanthrol*, m. 178-81° (from Et_2O), deep red (no yield given); *4-(2-naphthylazo)-2-phenanthrol*, red crystals (no m.p. given) (95.4%); *4-(1-naphthylazo)-2-phenanthrol*, m. 186-8° (from Et_2O), red crystals (88.1%); *1-(1-naphthylazo)-2-phenanthrol*, m. 228.9° (90.8%); *1-(2-naphthylazo)-2-phenanthrol*, gives a yellow color in dil. solns., no m.p. given (91.9%). 3-Phenanthrol (2 g.), 10 cc. 25% NH_4OH, and 4 g. $(NH_4)_2SO_3$ heated in a sealed tube for 28 hrs. at 210-20° gave 75.4% *3-phenanthrylamine*, m. 85°; HCl salt, no m.p. given. Diazotization of this in HCl and treatment with 2-naphthol in NaOH soln. gave *1-(3-phenanthrylazo)-2-naphthol*, red, no m.p. given; coupling with 3-phenanthrol as above gave *4-(3-phenanthrylazo)-2-phenanthrol*, red powder, does not m. 300° (from pyridine). C. M. Kosolapoff

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Condensation of tetraphenylbutynediol with phenol. Yu. S. Zal'kind, V. K. Teterin, and S. G. Kuznetsov (Leningrad Chem. Tech. Inst.). *J. Gen. Chem.* (U.S.S.R.) 15, 488-98(1945)(English summary).—[1CC(OH)-Ph$_2$]$_2$ (I) (30 g.), 37.5 g. PhOH, 60 cc. benzene and 0.4 g. dry $H_2NC_6H_4SO_3H$ were refluxed for 2 hrs. with continuous removal of water by means of a Stark-Dean type collector, in which 3.35 cc. H_2O was collected at the end of the reaction; after cooling and diln. with benzene, the cryst. and the liquid portions were steam-distd. to remove the solvent. There were obtained 28.7 g. cryst. matter and 16.7 g. red resin. Prolonged recrystn. from benzene, $CHCl_3$, and ligroin gave the following products: 21.5 g. *1-diphenylmethylene-2-p-hydroxyphenyl-3-phenylindene*, m. 255° (II), yellow-orange needles; 2.8 g. *1-diphenylmethylene-2-phenoxy-3-phenylindene* (III), m. 223°, orange prisms; 1.2 g. *2,2,5,5-tetraphenyl-3-phenoxy-2,5-dihydrofuran*, m. 170°, colorless needles; and 0.4 g. *2,2,3'-triphenyl-1',2',-3,4-indenochroman* (IV), m. 210-17°, colorless parallelopipeds. II was converted into the *MeO deriv.*, m. 170-7°, by boiling with MeI in the presence of K_2CO_3 in Me_2CO, or by treatment with Me_2SO_4 in 20% NaOH; rapid crystn. from Me_2CO leads to yellow needles of the above m.p., slow crystn. gives large red-brown parallelopipeds, m. 167-8°. III was prepd. by an alternate method for identification: 10.5 g. I in 150 cc. Et_2O was treated with 22.5 g. PCl_5 at 1° over 5 hrs., stirred for 2 hrs. at 0° and for 4 hrs. at room temp. to yield, after removal of the solvent, hydrolysis, and crystn. from ligroin 1.0 g. *1-diphenylmethylene-2-chloro-3-phenylindene*, m. 157° (cf. Wieland and Kloss, *C.A.* 23, 3696), and 3 unidentified products m. 147-8° (4.5 g.), m. 194-5° (0.2 g.), and m. 165° (0.4 g.); 0.4 g. of the chloride was added to a soln. of 0.1 g. K in 2 g. molten PhOH and heated slowly to 220° for 1 hr.; after treatment with alk. water, extn. of the ppt. with EtOH, and crystn. of the residue from benzene-petr. ether there was obtained a product identical with III, m. 223°, above. Boiling of this in MePh in the presence of sulfanilic acid failed to effect any isomerization. IV on boiling with alc. KOH is transformed into 2 substances which were unidentified: colorless, m. 250° (from EtOH, then EtOH-Me_2CO), and orange, m. 208-19°, with the former transforming into a product m. 208-14°, on heating above the m. p. The condensation of I with PhOH was also conducted in the presence of the following catalysts: activated Chasoviarskii clay, H_2SO_4-AcOH, and glacial AcOH. The 1st catalyst gave results similar to sulfanilic acid while the 2nd catalyst gave only II, m. 255°, in 98% yield (crude); the 3rd catalyst gave only a resinous, ill-defined mixt. of some transformation products of the glycol without condensation with PhOH. G. M. Kosolapoff

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Ca

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Isomerization of an alcohol with two double bonds. Yu. S. Zal'kind and A. I. Kulikov (Leningrad Chem. Tech. Inst.). *J. Gen. Chem.* (U.S.S.R.) 15, 643-9 (1945)(English summary).—The prepn. and hydrogenation of *phenyl(vinylethynyl)carbinol* (I) were studied. Hydrogenation results apparently in 1-phenyl-2,4-pentadien-1-ol, which isomerizes into *1-phenyl-1,4-pentadien-3-ol* (II) and *1-phenyl-1,3-pentadien-5-ol* (III). The starting material was prepd. conventionally from BzH and CH_2:CHC:CMgX, with the best (73%) yield resulting from addn. of BzH to the Mg compd. at −10° and letting the mixt. stand for 4-6 hrs. at −10°, and 12 hrs. at room temp. I, b_4 110°, b_5 125°, b_6 131°, b_8 137°, b_{10} 140°, b_{12} 142°, d_4^{20} 1.0453, n_D^{20} 1.57467. Hydrogenation of I in EtOAc in the presence of colloidal Pd gave the *diene alc.* mixt., b_4 111-12°, d_4^{20} 0.9924, n_D^{20} 1.54036; it remains quite unchanged on treatment with alc. KOH and its ozonization gave HCHO, BzH, cinnamic acid, and aldehyde. The diene alc. mixt. (8 g.) and 8 g. Cl_3CCO_2H in 8 cc. glacial AcOH, kept in a sealed tube for 22 hrs. at room temp. and for 4 hrs. at 100°, yielded 2 fractions of *crude trichloroacetate*, 0.3 g., b_5 94-105°, n_D^{20} 1.54052, and 0.7 g., b_5 104-5°, n_D^{20} 1.53211; better results were obtained when distn. was omitted. Hydrolysis of the crude ester, after washing with water, with alc. KOH at reflux yielded 3 fractions of III, 0.7 g., b_5 117°, 0.7 g., b_5 117-20°, n_D^{20} 1.52997, and 0.5 g., b_5 120°, formed apparently from isomerization during esterification. The isomeric mixt. of the diene alcs. from hydrogenation, however, consists principally of II, with only traces of the primary alc. Hydrogenation with addn. of 4 H resulted in formation of an isomeric mixt. of *olefinic alcs.*, $b_{1.5}$ 93-3°, d_4^{20} 0.9815, n_D^{20} 1.52561, which, on ozonization, gave BzH and BzOH.

G. M. Kosolapoff

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

A. E. Favorskii. Yu. S. Zal'kind. J. Applied Chem. (U.S.S.R.) 18, 881-90(1945).—Obituary with 30 references to scientific publications. G. M. Kosolapoff

COMMON ELEMENTS

COMMON VARIABLES

ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION

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CA

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Action of some inhibitors in catalytic hydrogenation in the presence of palladium. Yu. S. Zal'kind and Kh. V. Bal'yan. *Trudy Leningrad. Tekhnol. Inst. im. Lensoveta* **1946**, No. 12, 73-86.—The inhibiting action of NH_4CNS, $(NH_2)_2CS$, $Pb(OAc)_2$, and $Cu(OAc)_2$ during hydrogenation with colloidal Pd of ($:CC(OH)Me_2$) (I), $Me_2C(OH)C:CH$ (II), and $Me_2C(OH)CH:CH_2$ (III) in general decreases in the above order. The Pb salt is more active with alcs., and the Cu salt with the glycol. II is less affected by the inhibitors than I, but III somewhat more than II and less than I. Ethylenic alcs. absorb H fastest when *in statu nascendi* from the acetylenic ones. KCN, Na_3AsO_3, Na_2AsO_4, $CoSO_4$, and $NiSO_4$ are inactive as inhibitors. The inhibiting action is believed to be due to an interaction of the inhibitor with the org. compd. rather than with the Pd. Exptl. data are tabulated.

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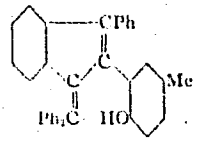
Condensation of *syn*-tetraphenylbutynediol with *p*-cresol. Yu. S. Zal'kind, V. K. Teterin, and S. G. Kuz-[illegible] (Leningrad Chem. Tech. Inst.). *J. Gen. Chem.* (U.S.S.R.) 16, 611-20(1946); cf. C.A. 40, 1705[illegible]. Tetraphenylbutynediol (I) (9.8 g.), 10.8 g. *p*-cresol, 26 cc. benzene, and 0.1 g. anhyd. sulfanilic acid were refluxed 5 hrs., using a reflux trap to collect the resulting water, of which 0.72 cc. was collected; the residual mass was distd. with steam and the residual red oil was dried by azeotropic distn. with benzene. On cooling the benzene soln. there was obtained 2.1 g. *1-diphenylmethylene-2-(p-tolyoxy)-3-phenylindene* (II), m. 218° (from benzene), while the benzene mother liquor on dilu. with ligroin gave a mixt. of 2 substances, sepd. by leaching with Et₂O, which left behind 2.5 g. *6-methyl-2,2,3'-triphenyl-1',2'-3,4-indenochroman* (III), m. 222-4° (from ligroin); repeated crystn. of the Et₂O-sol. material from benzene-ligroin gave 2.3 g. *2,2,3,5-tetraphenyl-3-(p-tolyoxy)-2,5-dihydrofuran*, m. 156° (from petr. ether); concn. of the mother liquor and prolonged standing gave 0.02 g. *tetraphenylbutatriene*, m. 236° (from benzene). The same products but in reduced amts. were obtained when the condensation reaction was run in the presence of activated clay instead of sulfanilic acid. The structure of II was confirmed by an independent synthesis in which 4 g. *p*-cresol was converted into the K deriv. and the latter was heated 1.5 hrs. to 230° with 1-diphenylmethylene-2-chloro-3-phenylindene in the presence of 0.02 g. powd. Cu; in addn. to a product, m. 218°, identical with II, above, there was obtained some *1-diphenylmethylene-3-phenylindene*, m. 204° (from Me₂CO). III (0.6 g.), 5 g. KOH, and 30 cc. 92% EtOH were refluxed 3 hrs. with stirring and the products were fractionally crystd. from ligroin, yielding a small amt. of an unidentified product, m. 215°, which did not have OH groups and which on heating above its m.p. was transformed to a compd. m. 177-80°, which was the principal product of the reaction and was identified as *1-diphenylmethylene-2-(4'-hydroxy-5'-methylphenyl)-3-phenylindene*, m. 177-80° (from ligroin); treatment with Me₂SO₄ in the presence of 20% NaOH gave the *Me ether*, m. 199° (from Me₂CO-petr. ether).



G. M. Kosolapoff

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ZAL'KIND, YU. S.

PA 27T4

USSR/Chemistry - Hydrofluoric Acid Nov 1947
Chemistry - Catalysis

"Anhydrous Hydrogen Fluoride as a Catalyst," Yu. S. Zal'kind, $\frac{1}{2}$ p

"Priroda" No 9

Article treats the use of hydrogen fluoride in the condensation of saturated hydrocarbon "isostructures" with olefin according to the equation:

$$C_mH_{2m+2} + C_nH_{2n} \rightarrow C_{m+n} + H_{2(m+n)+2}.$$

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CA

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Addition of hydrogen to acetylene derivatives. XXXVIII. Ethers of tetraphenylbutynediol and their catalytic hydrogenation. Yu. S. Zal'kind and P. S. Batsev (Pedagog. Inst., Leningrad). *J. Gen. Chem.* (U.S.S.R.) 17, 1358-67(1947)(in Russian); cf. *C.A.* 40, 3095[8].—$Ph_2C(OMe)C{:}CC(OMe)Ph_2$ (I) and the corresponding *EtO compd.* (II) were prepd. according to Arbuzov (*C.A.* 18, 2727). The *Pr compd.* (III) was prepd. by heating 10 g. $[Ph_2C(OH)C{:}]_2$ (IV) with 0.6 g. iodine and 115 cc. PrOH for 2 hrs. and cooling; III, m. 92-3°, sepd. on cooling. I with CrO_3 in AcOH gave a *diketone*, $C_{28}H_{20}O_4$, a dark oil, which gives a *semicarbazone*, m. 151-2°, and on heating with o-$C_6H_4(NH_2)_2$ in the presence of alc. HCl gives a *quinoxaline*, m. 172-3° (from EtOH); with excess CrO_3 in AcOH the ketone gave *methoxydiphenylacetic acid*, m. 99-100°. Similarly, II on oxidation gave a diketone, *1,1,4,4-tetraphenyl-1,4-diethoxy-2,3-butanedione*, a dark oil (*semicarbazone*, m. 168-9° (from EtOH); *phenylhydrazone*, m. 127-8°; *quinoxaline*, m. 182°), and on further oxidation gave *ethoxydiphenylacetic acid*, m. 114°. IV (6 g.), 2.7 g. iodine, 40 cc. benzene, and 20 cc. abs. EtOH boiled 5 hrs. gave 5.4 g. $[Ph_2C(OH)C{:}]_2$, m. 210° (from EtOH-ligroin), which reacts with EtMgBr and does not give C_2H_6 with KOH. To 5 g. I in 50 cc. $CHCl_3$ was added over 8 hrs. 4 g. Br in 35 cc. $CHCl_3$; after 7 hrs. heating to 45-50°, the mixt. was cooled to give 4.2 g. $[Ph_2(MeO)CCBr{:}]_2$, m. 196-7° (from ligroin). II similarly gave the *EtO analog*, m. 199-200°, and III gave the *PrO analog*, m. 195°. I (6 g.) in 70 cc. $CHCl_3$ treated with 4 atoms Cl and allowed to stand overnight gave 6.15 g. $[Ph_2(MeO)CCCl{:}]_2$, m. 194-5° (from Et_2O). IV (10 g.), 75 cc. benzene, and 1.12 g. NaOAc, treated in the cold with 23 g. Ac_2O, allowed to stand 0.5 hr., heated 2 hrs. to 45-50°, poured into water, gave no extn. with Et_2O 8.6 g. $Ph_2C(OAc)C{:}CC(OH)Ph_2$, m. 138°. In the presence of pyridine with excess AcCl there was obtained the *di-Ac deriv.*, m. 168-70° (from ligroin). I, II, and III failed to add H in the presence of Pd or Pt. The mono-Ac deriv. added 2 H at a rate slower than that of the free glycol in the presence of 5 mg. Pd per 0.01 mol. of the substance at atm. pressure; the reaction stopped after addn. of 2 H. The di-Ac deriv. failed to add H either with Pd or Pt catalysts. The *mono-Ac deriv.* added 4 H, with a Pt catalyst, giving after 19 hrs. (with 20 mg. Pt per 0.01 mole) the *tetrad. compd.*, m. 157° (from EtOH); the product of addn. of 2 H m. 149°.

G. M. Kosolapoff

ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION

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ZAL'KIND, Yu. S. DECEASED

12 to 15 May 1948, Moscow, first conference was held on history of Soviet chemistry, convened by Commission on the History of Chemistry, Acad. Sci. USSR. Many papers were presented by (ostensibly) members of this Commission.

"D. I. Mendeleyev's 'Organic Chemistry'--First Russian Text on Organic Chemistry" (Lled 23 Nov 1948)

"Materials on the History of Soviet Chemical Science," published by Acad Sci USSR in Moscow-Leningrad 1950.

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"APPROVED FOR RELEASE: 09/19/2001 CIA-RDP86-00513R001963710009-5

ZAL'KIND, YU. S.

"Synthesis of Long Chains of the Polypeptide Type," Priroda, No. 2, 1948.

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"APPROVED FOR RELEASE: 09/19/2001 CIA-RDP86-00513R001963710009-5

ZAL'KIND, YU. S.

"Light Sensitive Glass," Priroda, No. 5, 1948. Prof.

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"APPROVED FOR RELEASE: 09/19/2001 CIA-RDP86-00513R001963710009-5

ZAL'KIND, YU. S. PA 9/49T43

USSR/Chemistry - Aldehydes
Chemistry - Synthesis

Sep 48

"New Method for Synthesizing Aldehydes," Yu. S. Zal'kind, 1 p

"Priroda," No 9

Discussion of synthesis of acids from unsaturated compounds of hydrogen oxides and water was published in "Priroda" No 3, 1947. Further research reveals that if unsaturated derivatives and hydrogen oxides are treated with hydrogen in presence of a catalyst and heated, aldehydes can be produced (US Patent 2327066). Traces chemical process from formation of ethylene to final product--aldehyde.

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"APPROVED FOR RELEASE: 09/19/2001 CIA-RDP86-00513R001963710009-5

ZAL'KIND, YU. S.

"Metallic Titanium and Zirconium," Priroda, No. 10, 1948.

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"APPROVED FOR RELEASE: 09/19/2001 CIA-RDP86-00513R001963710009-5

ZAL'KIND, Yu. S. (Prof)

USSR/Nuclear Physics -- Radioactivity Dec 48
Chemistry -- Periodic System

"Review of A. E. Fersman's Book, 'The Role of Mendeleyev's Periodic Law in Contemporary Science,'" Prof Yu. S. Zal'kind, 1 p

"Priroda" No 12

Brochure is series of lectures given by Fersman during 1946, with well-organized and thorough treatment of subject. Main consideration is revolution which occurred in chemistry with discovery of radioactive substances. Recommended reading for the expert and for the general public. Moscow, 1947, 56 pages, price 2 rubles, 40 kopecks.

[redacted] 25/49T87

[redacted] 25/49T87

PA 25/49T87

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Addition of hydrogen to acetylene derivatives. XXXVIII. An erythritol of the diacetylene series and its catalytic hydrogenation. Yu. Zal'kind and L. Zhurav-leva. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 18, 984-9 (1948); cf. *C.A.* 42, 4161c.—EtMgBr (from 50 g. EtBr) and 10 g. $Me_2C(OH)C{\cdot}CH$ in Et_2O were allowed to react with cooling and after standing overnight were treated with 12 g. benzil in Et_2O, stirred several hrs., and allowed to stand 2 days, yielding cryst. $[Ph(HO)CMe_2C{\cdot}C{\cdot}C(OH)]_2$, 66.6%, m. 144-5° (from EtOH), best sol. in Me_2CO, less sol. in EtOH or EtOAc; heating with EtOH-Na yields C_2H_2; addn. of Br in $CHCl_3$ was unsuccessful; oxidation by $KMnO_4$ gave $Me_2C(OH)CO_2H$ and B_2OH, as well as a ketone (semicarbazone, m. 181-2°). The product (3 g.) and 10 g. Ac_2O in the presence of a little H_3PO_4 gave in 10 days at 20-5° 55.4% cryst. *tetraacetate*, m. 150-3° (from Me_2CO); the alc. with Ac_2O in the presence of NaOAc and pyridine gave the *diacetate*, m. 132-3°. Hydrogenation of the alc. in EtOAc over Pd is very slow after the addn. of sufficient H to convert the triple bonds to double bonds (essentially stopped after 30 min.), yielding *2,9-dimethyl-5,6-diphenyl-3,7-decadiene-2,5,6,9-tetrol* (I), m. 146.5-47°. Similar hydrogenation over Pt black gives rapid and complete reaction, yielding the product as a noncrystallizable oil after addn. of 8 H atoms per mole. Hydrogenation of the tetraacetate with a Pd catalyst gave rather more rapid reaction than with the alc. itself, yielding the corresponding I *tetraacetate*, m. 148-51°, although after addn. of 4 H atoms the reaction still showed signs of slow progress. With a Pt catalyst the addn. of H is complete and proceeds further, apparently by replacement of the ester groupings by H, yielding hydrocarbons; this product was a noncryst. oil.

G. M. Kosolapoff

ASM-SLA METALLURGICAL LITERATURE

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PA 6/49T1

ZAL'KIND, YU. S.

May 48

USSR/Chemistry - Organic Compounds
Chemistry - Halogenation

"The Action of Bromine and Chlorine on a Glycol of the Diacetylene Series," Yu. S. Zal'kind and N. N. Mel'teva, Lab of Org Chem, Leningrad Technological Inst imeni Lensovet, 10½ pp

"Zhur Obshch Khim" Vol XVIII (LXXX), No 5

Studies the action of bromine and chlorine on 2, 5-dimethyl-octa-3, 5-diene-2, 7-diol, which produces a complex mixture of products. When they are halogenized by a 10% alkali solution, 3, 4-dichlor and 3, 4-dibrom-5-keto-2, 5-dihydrofuran is obtained. Submitted 13 Sep 1947.

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Action of bromine and chlorine on glycols of the acetylene series. Yu. S. Zal'kind and N. N. Mel'teva. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 18, 990–1000(1948)(in Russian). 2,5-Dimethyl-3,5-octadiyne-2,7-diol (I) with Cl or Br gives a complex mixt. of products. When I (m. 132.5°; cf. Z. and Afrikovich, C. A. 31, 4284) (2.7 g.) in 50 ml. $CHCl_3$ was treated at −2° to −5° with 0.80 ml Br, no HBr evolution was noted, but on attempts to remove the solvent by mild heating the product lost HBr and formed a black mass; removal of solvent *in vacuo* without heating gave viscous, yellow, liquid *dibromide isomers*, which could not be distd. A similar reaction, but using 4 atoms Br at 0–12°, led to partial loss of HBr in 2–48 hrs. standing, and the removal of the solvent *in vacuo* gave an undistillable residue, apparently a mixt. of *tetra-* and *hexabromides*. When 10 g. I in 50 cc. $CHCl_3$ was treated with 39.6 g. Br, the residue, after the removal of solvent, gave 1.2 g. solid, m. 167.9° (from EtOAc), corresponding to $C_{10}H_{12}OBr_7$; this substance is better obtained by the direct action of Br vapor on I; it is given the provisional formula: $CBr:CBr.CMe_2.O.CBrCBr_2CBr_2CBrMe$-$CH_2Br$, since on oxidation by Cr oxide in AcOH it yields a *product*, $C_8H_8Br_4O_2$, m. 129–9.5°, which is also obtained by the action of 10% NaOH on the crude bromide. Small amts. of Br-contg. products, m. 101–3°, 137.8°, and 70.8°, were obtained but were not identified. The heptabromide is quite resistant to hydrolysis by 0.5 N alc. KOH, as no reaction occurs in 24 hrs. at room temp. and only a slow reaction at 50°; hydrolysis is 85%, complete in 12 hrs. at 80–90°. Addn. of 2.5 g. Cl to 5 g. I in $CHCl_3$ at 5–6° yields 2.4 g. unreacted I and an oil, b_6 64–130°, which loses HCl slowly on distn., and appears to be a *dichloride*; when 4 atoms Cl were used, 5.0 g. I gave 5 g. tar and 2.0 g. oil, b_6 68–100°, apparently a *di-* and *tetrachloride mixt.* Use of 8 atoms Cl gave an undistillable yellow oil mixt. of *tetra-* and *hexa-Cl derivs.* An excess of Cl passed at 0° through I in $CHCl_3$ gave a mixt. of products which decompd. on distn., and only 1 definite product, b. 52–7° *in vacuo* (not specified), m. 42–3°, was obtained; this was $C_2H_4OCl_2$, identified as *dichloroacetone*; washing the product with H_2O and 10% NaOH gave a slowly crystg. oil, m. 81° (from ligroin), identified as *2,2-dimethyl-3,4-dichloro-5-keto-2,5-dihydrofuran*; the crude chlorination product oxidized with $KMnO_4$ gives dichloroacetone, HCO_2H, and the above ketofuran. The formation of the furan derivs. is believed to take place through the cyclization of the α,β-di-Cl ketone intermediate. Possible reaction mechanisms are discussed. G. M. K.

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