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Freydlina, R. Kh.

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FREYDLINA, R. Kh.

BC

Arylation of mercuri-organic hydroxides by means of organic derivatives of tin, antimony, and boron. R. C. FREIDLINA, A. A. KOTSCHESCHKOV, and A. N. NESMEJANOV (J. Gen. Chem. Russ., 1936, 6, 1171—1175).—The following compounds of the type HgRR' have been prepared by the general reaction $RHgX+R'_nMX_n \rightarrow HgRR'+R'_{n-1}MX_{n-1}$ (M=Sn, Sb, or B; X=halogen or OH): R=Me, R'=α-$C_{10}H_7$, m.p. 80—82°, from RHgI and R'_2SnCl_2, in EtOH–NaOH at the b.p.; R=Ph, R'=m-, m.p. 113—116°, or p-$C_6H_4NO_2$, sintering at 144—145°, from m- or p-R'HgCl and R_2SnCl_2, as above; R=p-C_6H_4Cl, R'=CH_2Ph, m.p. 104—106°, from R'HgCl and R_3SbO.

R. T.

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PROCESSES AND PROPERTIES INDEX

a-3

(A) Structure of products of addition of mercury salts to unsaturated compounds by the arylation method. A. N. NESMEJANOV and R. C. FREIDLINA. **(B) Reaction of dimethylethane with β-bromomercuriethyl alcohol, and the structure of the products of addition of mercuric salts to olefines.** R. C. FREIDLINA, A. N. NESMEJANOV, and F. A. TOKAREVA (J. Gen. Chem. Russ., 1937, 7, 42—50, 262—266).—(A) OH·CH$_2$·CH$_2$·HgBr (I) in C$_6$H$_6$ and PhNCO yield *β-bromomercuriethyl phenylcarbamate* (II), m.p. 124—126° (decomp.). (I) in aq. alcoholic KOH and di-*p*-tolyldichlorostannane afford *β-hydroxyethyl-p-tolylmercury* (III), m.p. 52·5—53·5°. Hg(OAc)$_2$ and cyclohexene (IV) in H$_2$O yield *2-acetomercuricyclohexanol*, m.p. 112·5—113·5°, which reacts with SnPh$_2$Cl$_2$ (V) in EtOH-KOH, at the b.p., to afford *2-phenylmercuricyclohexanol* (VI), m.p. 101—102°. Hg(OAc)$_2$ and (IV) in EtOH give *1-ethoxy-2-acetomercuricyclohexane*, m.p. 70°, converted by boiling with NaOH and (V) in EtOH into *Hg phenyl 2-ethoxycyclohexyl* (VII). 1-Chloromercurimethyl-1:2-dihydrobenzfuran, NaOH, and (V) in EtOH, at the b.p., afford *1-phenylmercurimethyl-1:2-dihydrobenzfuran*, m.p. 60—61°. This, similarly to (II), (III), (VI), and (VII), is decomposed by 15% HCl, with production of unsaturated hydrocarbon and Hg aryl chloride. The reactions support the structure given above for (I), rather than one involving residual valencies, of the type C$_2$H$_4$·HgBrOH. (B) (I) and CH$_2$N$_2$ in Et$_2$O yield β-bromomethylmercuriethyl alcohol, which decomposes at room temp. with production of C$_2$H$_4$. *Hg, bromomethylmercuric bromide* (VIII), m.p. 124—125°, CH$_2$O, and N$_2$. HgBr$_2$ and CH$_2$N$_2$ in Et$_2$O yield (VIII) and *Hg dibromodimethyl*, m.p. 42—43°. (VIII) and aq. NaOH yield Hg, CH$_2$O, and HBr. R. T.

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FREIDLINA, R. KH.

"Sur la reaction du diazomethane avec l'etanolmercuro-bromure et sur la structure des produits de la dixation des sels sur mercure sur les olefines."
Freidline, R. Ch., Nesmelanow, A. N. et Tokarewa, F. A,. (p. 262.)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii) 1937, Volume 7, No. 1

On the reaction of diazomethane with ethanolmercuric bromide and on the structure of the products of fixation of mercury salts on olefines.

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

Mercury organic compounds. XV. The structure of the addition compounds of mercury salts with styrene and α-methylstyrene. A. N. Nesmeyanov and R. Kh. Freidlina. *J. Gen. Chem.* (U. S. S. R.) 7, 2748-53(1937); cf. *C. A.* 30, 8150°. —When equimol. amts. of styrene and $Hg(OAc)_2$ react in H_2O soln., $PhCH(OH)CH_2HgOAc$ (I), m. 77-9° (decompn.), is formed. With Na-Hg this is reduced to PhCH(OH)Me. I and NaCl form the corresponding *mercurichloride*, m. 95-6°, and with NaBr it gives the *bromide*, m. 102-3°. When excess Hg salts are used, compds. of the type of $3(PhCH(OH)CH_2HgCl)_2HgCl_2$ are formed. $PhC(CH_3):CH_2$ and $Hg(OAc)_2$ give $PhC(OH)(CH_3)CH_2HgOAc$ (II), m. 101-2°, which is reduced to $PhC(CH_3)_2CH_2OH$. The *chloride* of II is a liquid, and the *bromide* m. 45-6°. The compd. obtained by Manchot (*C. A.* 15, 508) from styryl Et ether and $Hg(OAc)_2$ probably has the structure PhCH(HgOAc)CH(OH)OEt.

H. M. Leicester

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

CA

10

Structure of addition products of metal halides and unsaturated compounds. R. Kh. Freidlina and A. N. Nesmeyanov. *Compt. rend. acad. sci. U. R. S. S.* 26, 60-4(1940)(in English). The addn. product of $HgCl_2$ and C_2H_2, $C_2H_2 \cdot HgCl_2$, can be prepd. easily by satg. (for 3 hrs.) a soln. of 72 g. $HgCl_2$ in 75 ml. HCl (1:1) with C_2H_2. The yield is 25-30 g.; m. p. 123-4° (decompn.). Addn. of NH_3 to a $CHCl_3$ soln. of this product yielded $(C_2H_2)_2HgCl_2$ which, after recrystn. from a mixt. of petr. ether and ether, m. 59-60°. These compds. react with Ph_4SnCl_2 to form C_2H_2, PhHgCl and $SnCl_2$; with CH_3N_3 to form $ClCH_2HgCl$, N and C_2H_2; and with Ph_3P to form C_2H_2 and $(Ph_3P)_2HgCl_2$. These products are the same as those formed by permitting free $HgCl_2$ to react with the resp. reagents. Acting as true organomercuric compds. these substances also react with I in ether to yield HgCl + ClCH:CHI. This quality in chem. behavior is explained by partial dissocn., tautomerism of the at. and mol. forms, and a hybrid-resonance: ClCH:CHHgCl ⟷ $C_2H_2HgCl_2$. A study of the Raman spectra is proposed.

H. A. Frediani

Lab. Organometallic Compounds. Inst. Org. Chem., AS USSR

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

CA

10

Synthesis of diaryliodonium salts. R. Kh. Freidlina and A. N. Nesmeyanov. *Compt. rend. acad. sci. U. R. S. S.* 29, 567-70(1940)(in English).—Aromatic organometallic compds. of Sn are the best arylation agents. Reactions with these compds. proceed under mild conditions, often at room temp. in good yields. For example, $PhSnCl_3$ reacts with ICl_3 in dil. HCl energetically, with liberation of heat, giving a quant. yield of Ph_2ICl, thus arylating ICl_3 as well as $HgCl_2$ according to the equation. $ICl_3 + 2PhSnCl_3 \rightarrow Ph_2ICl + 2SnCl_4$. To 2 g. (0.008 mol.) ICl_3 in 10 cc. dil. (1:10) HCl was added 5 g. (0.017 mol.) $PhSnCl_3$. After 0.5 hr. the reaction mixt. was cooled, the ppt. sepd. and decompd. by heating with boiling H_2O. After filtration and cooling, 0.8 g. Ph_2ICl sepd. Evapn. of the mother liquor yielded an addnl. 1.15 g. of product, or a total yield of 72%. After repptn. from H_2O it m. 220°, with conversion into a mixt. of PhCl and PhI. $PhICl_2$ can be arylated very readily in good yields with $PhSnCl_3$ at room temp., $PhICl_2 + PhSnCl_3 \rightarrow Ph_2ICl + SnCl_4$, under exptl. conditions analogous to those described in the previous synthetic method. The latter method can be used as well for the synthesis of aryl iodochlorides and asym. iodonium salts of the type RR'ICl. With Ph_2Hg and ICl_3, Ph_2ICl and $HgCl_2$ were obtained. The yield is somewhat lower in the latter reaction, because PhIO is obtained as a by-product. The product from the latter reaction m. 168-170° and is believed to be a double salt in agreement with analysis, $Ph_2ICl.HgCl_2$ (51% yield). This new method of synthesis of iodonium salts has the advantage of simplicity and shortness compared to the methods previously described in the literature.

W. A. Cook

Lomonosov Inst. Fine Chem. Tech., Chair Org. Chem., Moscow

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FREYDLINA, R. Kh.

"On the Organometallic Compounds of Mercury: XXI. On the Properties and the Structure of Mercuric Derivatives of Acetylene," Iz. Ak. Nauk SSSR, Otdel. Khim. Nauk, No.1, 1942

Chair Organic Chem., Moscow Inst. Fine Chem. Tech. im. Lomonosov

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

Ca

10

Addition of ICl_3 to C_2H_2 and structure of 2-chlorovinyl iodochloride. A. N. Nesmeyanov and R. Kh. Freidlina. *Bull. acad. sci. U. R. S. S., Classe sci. chim.* 1943, 152-5.—See *C. A.* 37, 868[6]. O. Berend

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1ST AND 2ND ORDERS

3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

Ca

10

Freidlina, M. Kh.: Synthetic Methods in the Field of Metallo-organic Compounds of Arsenic. (In Russian.) Izdatel'stvo Akademii Nauk SSSR. 1945. 180 pp. R. 3.

COMMON ELEMENTS

COMMON VARIABLES INDEX

OPEN

MATERIALS INDEX

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1ST AND 2ND ORDERS — 3RD AND 4TH ORDERS

PROCESSES AND PROPERTIES INDEX

10

Organometallic compounds of mercury. XXVII. Interaction of olefins with mercuric salts in aprotic media. R. Kh. Freidlina and N. S. Kochetkova (Inst. Org. Chem., Acad. Sci., U.S.S.R.). *Bull. acad. sci. U.R.S.S., Classe sci. chim.* 1945, 128-36(in English, 136); cf. *C.A.* **39**, 1637°.—The reaction of C_3H_6 with $HgCl_2$ in piperidine has been investigated. An opinion about the structure of the product is given. Dry $HgCl_2$ (7 g.) and 130 cc. of dry piperidine were shaken for 45-50 hrs. in a vessel filled with C_3H_6 under pressure of 35-40 cm. of Hg. The ppted. crystals of $C_5H_{10}.NH.HCl$, filtered off, washed with ether, and recrystd. from abs. alc., m. 243°; yield 73.8%. The filtrate, evapd. under vacuum without admitting CO_2 or atm. moisture and without heating, yielded 60.5% of crude $C_5H_{10}NCH_2CH_2HgCl$ (I), m. 35-40° which, when dried over H_2SO_4 under vacuum and recrystd. from a mixt. of abs. alc. and ether, m. 77.5-8°, decomps. 124°. It is a white, fine, cryst. powder, very sol. in $CHCl_3$ and benzene, sol. in water, alc., slightly sol. in ligroin and petroleum ether, reduced with Na-Hg to 1-ethylpiperidine, recovered as a picrate, m. 167° (yield 25%). I with a satd. soln. of KI gave quantitatively $C_5H_{10}NC_2H_4HgI$ (II), m. 100-1° (decompn.) after purification, white, shiny platelets, sol. in alc., ether, $CHCl_3$, and benzene, insol. in water. I with dry HCl in ether gave the HCl salt, which after recrystn. from abs. alc., m. 132° (decompn.), white shiny needles, very sol. in H_2O and aq. alc., insol. in ether, $CHCl_3$, and benzene. I in water treated with dil. or concd. caustic alkali or satd. with H_2S does not form a ppt. of HgO or HgS, resp. I and II readily give up C_2H_4 on heating or on treatment with an excess of KI or KCN. I (0.11 g.) in alc. treated with NaI, dild. with water after 30 hrs. and satd. with H_2S, ppted. 0.0723 g. of HgS. The thermal decompn. of I.HCl was studied; 0.1469 g. of the salt gave off 9.4 cc. of C_2H_4 (746 mm., 21.5°) on heating for 2 hrs. at 136-38°; 0.0964 g. heated 1 hr. at 130-6° in a stream of CO_2 evolved 4.8 cc. of C_2H_4 (754 mm., 23°) (76.33%); 0.0907 g. heated 1 hr. under the same conditions in a stream of dry HCl instead of CO_2 gave 3.3 cc. of C_2H_4 (752 mm., 23°) (40%). The mineral acid salts of aminoalkylmercuri compds. do not decomp. even on boiling with concd. mineral acids. 22 references. **XXVIII. Addition products of metal halides to unsaturated compounds.** A. N. Nesmeyanov, R. Kh. Freidlina, and A. E. Borisov. *Ibid.* 137-44(in English, 145).—The structure and the configuration of *cis*- and *trans*-2-chlorovinylmercuric chloride have been studied. The authors ascribe to the addn. product of C_2H_2 and $HgCl_2$ described by Biginelli (*Am farmacoterap. e chim.* 1890, 16-20) (m. 124°, soly. in CCl_4 0.65%, dipole moment 1.64D) a trans configuration and to its geometric isomer described by Nesmeyanov and Borisov (Part XXIX) (m. 79°, soly. in CCl_4 10%, dipole moment 2.76D) a cis configuration. They explain the quasicomplex behavior of the adducts of C_2H_2 and $HgCl_2$ by resonance, the superposition of the covalent structure and of the ionic one in which C_2H_2 is not connected with Cl^- and Hg^+Cl. The weight of the ionic structures was

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

Be

a-3

[illegible] A. N. Nesmejanov and R. C. [illegible] (Bull. Acad. Sci. U.R.S.S., Cl. Sci. Chim., 1946, [illegible]) [illegible] $HgCl_2$ (72 g.) in 75 ml. of 15% aq. HCl gives with C_2H_2 after 2 hr. at room temp., [illegible] g. of trans-CHCl:CH-HgCl (I), m.p. [illegible] (decomp.); a further yield is obtainable by continuing the action of C_2H_2 after adding more $HgCl_2$. (I) with NH_3 in C_2H_2 gives, in 10 min., [illegible] and $Hg(CH:CHCl)_2$ (II), m.p. 72° [illegible] (J.C.S., 1931, 158, 767), who gives m.p. 60°; [illegible] of his prep. gives a product with m.p. 60—70°. (II) with $HgCl_2$ in Et_2O gives (I), and with HgI_2 in Et_2O gives, in 2 hr., β-chlorovinyl [illegible], m.p. 116—[illegible]° (decomp.), which may also be obtained, in 2 hr., from 0·0007 mol. of (I) in 40 ml. of abs. EtOH with 0·013 mol. of NaI in 5 ml. of abs. EtOH. G. W.

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FREYDLINA, R. KH.

Ca

10

PROCESSES AND PROPERTIES INDEX

The synthesis and structure of bis(chlorovinyl)iodonium and phenyl(chlorovinyl)iodonium salts. R. Kh. Freidlina, K. M. Brainina, and A. N. Nesmeyanov (Inst. Org. Chem., Acad. Sci. U. S. S. R.). *Bull. acad. sci. U. R. S. S. Classe sci. chim.* 1943, 647-53(in English, 654).—When *trans*-ClCH:CHHgCl is treated with ICl_3 in 3% CCl_4, it gives 6% $(ClCH{:}CH)_2ICl.3HgCl_2$ (I), *m.* 149-50°. The low yield is due to the chlorinating action of ICl_3. I and H_2S give free *bis(2-chlorovinyl)iodonium chloride* (II), *m.* 202° (decompn.). When ICl_3 is treated with C_6H_6 and the product stirred with PhS_2Cl_2 it gives 31% *(2-chlorovinyl)phenyliodonium chloride* (III), *m.* 220-30° (decompn.) (*$HgCl_2$ double salt*, *m.* 135°). When II is heated above 185° and III above 220° they each lose about 30% C_2H_2. The $HgCl_2$ double salts are more stable. At 100°, II and III lose 5-10% C_2H_2 in C_5H_5N, and 20% C_2H_2 in 15% HCl. The free bases are even less stable, since boiling the compds. in H_2O causes loss of 12% C_2H_2, and, when alkalies are added, this rises to 30% even at room temp. These reactions show that II and III are quasicomplex compds. H. M. Leicester

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1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

CA

Reaction of aryl iodochlorides with 2-chlorovinylmercury chloride. E. M. Bralnina and R. Kh. Freidlina. *Bull. acad. sci. U.R.S.S., Classe sci. chim.* 1947, 623-30 (in Russian).—Reactions of $ArICl_2$ with *trans*-ClCH:CHHgCl were investigated. Introduction of a Me group into the Ph ring facilitated the exchange reaction, while introduction of NO_2 suppressed it, as may be expected on the basis of electron displacements caused by these electron-repelling and electron-attracting groups. The reactions of $PhSnCl_3$ with $ClCH:CHICl_2$ and $ICH:CHICl_2$ were also examd.; in the 1st instance $SnCl_4$ and ClCH:CHPhICl were formed (*C.A.* 40, 4080[9]); in the 2nd instance the products were Ph_2ICl, C_2H_2, and $SnCl_4$. This is explained by the +T effect for iodine being smaller than that of Cl. $PhICl_2$ (15 g.) and 15 g. *trans*-ClCH:CHHgCl (I) in 40 ml. 3% HCl, shaken for several hrs. at room temp. until the KI test was neg., gave 9 g. (31%) ppt. which, after washing with $CHCl_3$, m. 133-4° and was identified as *Ph(ClCH:CH)ICl.HgCl₂*, m. 135-7° (from dil. EtOH). Treatment with H_2S, filtration, and evapn. gave *phenyl(2-chlorovinyl)iodonium chloride*, decomp. 224-6°; *picrolonate* m. 158.5-9° (from dil. EtOH); *picrate* m. 167° (decompn.; from dil. EtOH). I (17.5 g.) and 17 g. *o*-$MeC_6H_4ICl_2$ similarly gave (*o*-MeC_6H_4)(*ClCH:CH)ICl.HgCl₂*, m. 98-9° (from H_2O), 40%; the *iodonium chloride* itself decomp. 202-3.5° (from H_2O); *picrolonate*, decomp. 102-2.5° (from dil. EtOH). I (11.3 g.) and 11 g. *p*-$MeC_6H_4ICl_2$ similarly gave (*p*-MeC_6H_4)(*ClCH:CH)ICl.HgCl₂*, decomp. 151-2° (from dil. EtOH); the *iodonium chloride*, decomp. 214-15° (from H_2O). I (40.6 g.) and 42.6 g. *p*-$ClC_6H_4ICl_2$ were shaken in 50 ml. 3% HCl 20 hrs. at room temp. without action; on heating to 70° the reaction was complete in 20-30 min., giving 5 g. (6%) (*p*-ClC_6H_4)(*ClCH:CH)ICl.HgCl₂*, decomp. 142-4° (from dil. EtOH); the *iodonium chloride*, decomp. 187-8° (from H_2O). Shaking 40 g. *m*-$NO_2C_6H_4ICl_2$ and 36 g. I in 100 ml. 3% HCl 4 hrs. at room temp., followed by sepn. of the pptd. oil and solid, gave, on distn. of the low-boiling halides on a steam bath, 40% *trans*-dichloroethylene, b. 48°, n_D^{20} 1.4465; the residue was distd. with steam, giving 81% *m*-$IC_6H_4NO_2$; the aq. soln. from the reaction mixt. yielded 85% $HgCl_2$, while the $CHCl_3$-insol. residue of the reaction mixt. was shown to be *m*-$O_2NC_6H_4$(*ClCH:CH)ICl.2HgCl₂*, decomp. 146-7° (from dil. EtOH) (5%); the *iodonium chloride* decomp. 170-2° (from H_2O). Under similar conditions, the *o*- and *p*-isomers do not react with the formation of double

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salts. Satn. of *trans*-diiodoethylene in $CHCl_3$ with Cl with ice-cooling gave 2-iodovinyl iodoilchloride, which was filtered off and washed with cold $CHCl_3$; when 3.0 g. of this and 4 g. $PhSnCl_3$ were stirred with cold 15% HCl for many hrs. until the reaction was complete and the ppt. was extd. with hot H_2O, the ext. gave, on cooling, 0.9 g. Ph_2ICl, decomp. 224–6° (from H_2O); evapn. of the filtrate gave an addnl. amt. of the product (total yield, 50%); *picrolonate* decomp. 178.5–9.5° (from dil. EtOH). All the aryl(2-chlorovinyl)iodonium chlorides obtained above eliminate C_2H_2 on treatment with dry pyridine, with 10–30% yields. Use of 40% NaOH raises the yield to 54–9% with the *o*-tolyl and *m*-nitro compds. $ICH:CHICl_2$ with 40% NaOH gives substantial amts. of C_2H_2, while pyridine raises the yield to 50–60%.

G. M. Kosolapoff

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PROCESSES AND PROPERTIES INDEX

B

25

*516. **Interaction of Acetylene and Mercury Chloride in the Absence of Solvent.** (In Russian.) R. Kh. Freidlina and O. V. Nogina. *Bulletin of the Academy of Sciences of U.S.S.R., Section of Chemical Sciences*, no. 1, 1947, p. 105-106.

The interaction of acetylene and mercury chloride in the absence of a solvent, at 100°C., results in formation of cis betachlorvinylmercuric chloride in good yield. The trans compound is formed only in a very small quantity.

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FREYDLINA, R. Kh.

"On Organometallic Compounds of Mercury. XXXVIII. On the Peculiarity of the Chemical Conduct of B-Aminoalkylmercuric Salts," Iz. Ak. Nauk SSSR, Otdel. Khim. Nauk, No.4, 1947

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FREYDLINA, R. KH.

USSR/Chemistry - Organic Compounds
Chemistry - Lead Compounds

Jan/Feb 1948

"Quasi-Complex Lead Compounds," A. I. Nesmeyanov, R. Kh, Freydlina, A. Kochetov, Inst of Org Chem, Acad Sci USSR, 7 pp

"Iz Ak Nauk SSSR, Otdel Khim Nauk" No 1

Describes how lead organic compounds belonging to the B -vinyl chloride group are obtained. Gives details of research on the interaction of these compounds with mercury and chloride of tin, and on their reaction to the action of alkalis, water, Grignard reagent, and hydrazidine.

P A 66T27

APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3"

"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R. KH.

PA 8/49T19

USSR/Chemistry - Thallium Compounds Jul/Aug 48
Chemistry - Organic Compounds

"Quasi-Complex Compounds of Thallium," R. Kh. Freydlina, Aleksandr Kochetkov, A. N. Nesmeyanov, Inst Org Chem, Acad Sci USSR, 5 3/4 pp

"Iz Ak Nauk SSSR, Otdel Khim Nauk" No 4

Describes preparation of thallium organic compounds containing a β-chlorvinyl group and investigates their reaction with mercury and stannous chloride. Studies reaction of di-β-chlorvinylthallous chloride to heat, alkali, mineral acid, Grignard's reagent and KI. Submitted 22 Dec 1947.

[redacted] 8/49T19

APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3"

"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R. KH.

PA 27/49T33

USSR/Chemistry - Organic Compounds, of Mercury Sep/Oct 48

Chemistry - Mercury Salts, Reaction With Carbon Monoxide

"The Field of Mercury Organic Compounds: XLI, Reaction of Carbon Monoxide With Mercury Salts in an Amine Medium," R. Kh. Freydlina, E. I. Kan, Inst Org Chem, Acad Sci USSR, 6 pp

"Iz Ak Nauk SSSR, Otdel Khim Nauk" No 5

Shows that carbon monoxide is easily joined with mercury salts in piperidine medium with the

[redacted] 27/49T33

USSR/Chemistry - Organic Compounds, of Mercury (Contd) Sep/Oct 48

formation of products of the following composition:

$$C_5H_{10}N-\underset{\underset{O}{\|}}{C}-H_g X \cdot C_5H_{10}NH.$$

[redacted] 27/49T33

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R. KH.

Aleksandr Nikolaevich Nesmeyanov. R. Kh. Freidlina, M. I. Kabachnik, and V. V. Korshak. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1949, 502 ff. — A review of his scientific work on his 50th birthday.
G. M. Kosolapoff

APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3"

"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

CA

12

1,6-Addition of mesitylmagnesium bromide to dimethylvinylcarbinol acetate. A. N. Nesmeyanov, R. Kh. Freidlina, and A. K. Kochetkov (Inst. Org. Chem., Moscow). *Izvest. Akad. Nauk S.S.S.R., Otdel Khim. Nauk* **1949**, 623-30.—$CH_2:CHCMe_2OAc$ (I), b. 121-2.5°, n_D^{20} 1.4118, d_4^{20} 0.8959, was obtained in 45% yield by heating 70 g. carbinol, 70 g. dry AcONa, and 100 g. Ac_2O 15 hrs. to 90-100°; a by-product, *3-methyl-2-butenyl acetate*, b. 151-2°, n_D^{20} 1.4283, d_4^{20} 0.9240, also forms. Hydrogenation of I over Pd-$CaCO_3$ gave *AcOCMe$_2$Et*, b. 123-4°, d_4^{20} 0.8770, n_D^{20} 1.4020. I (49 g.) and 2,4,6-$Me_3C_6H_2MgBr$ (from 100 g. $Me_3C_6H_2Br$) in Et_2O gave after refluxing 30 hrs. 37.5% mesitylene and 19.5 g. $C_{16}H_{24}$, b_{10} 143-4.5°, n_D^{20} 1.5100, d_4^{20} 0.9042, identified as *1-mesityl-3-methyl-2-butene*, which with H over Pt in AcOH gives the *satd. deriv.*, b_{10} 134-5°, while bromination in CCl_4 gives the *dibromide*, m. 68-9°.

G. M. Kosolapoff

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R. Kh.

A

Action of bromine on alkyl chlorocarbonates. R. Kh. Freidlina, O. V. Nogina, and A. N. Nesmeyanov (Inst. Org. Chem., Moscow). *Izvest. Akad. Nauk S.S.S.R., Otdel Khim. Nauk* 1949, 631-5.—Heating 90 g. $PrOCOCl$ with 132 g. Br 18 hrs. on a steam bath gave 20.5 g. $Br\cdot CH_2CH_2Br$, av. yield, 10-15%. Similarly, $PrOCOCl$ (40 g.) gave 15.0 g. 1,2-dibromopropane, b_{16} 32-3°, n_D^{20} 1.5192, d_4^{20} 1.9245 (24%); $BuOCOCl$ gave 43% $MeCHBr\cdot CHBrMe$, b_{16} 51-2°, b. 161°, n_D^{20} 1.5150, d_4^{20} 1.7892, iso-$BuOCOCl$ gave 40% mixed 1,2-dibromoisobutane and 1,2,3-tribromo-2-methylpropane, resp., b_{16} 62-3°, n_D^{20} 1.5088, d_4^{20} 1.7628, and b_{16} 105-6°, n_D^{20} 1.5670, d_4^{20} 2.1035.

G. M. Kosolapoff

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

Some new possibilities of synthesis of metalloorganic compounds of lead, tin, and thallium. A. K. Kochetkov and R. Kh. Freidlina (Acad. Sci. U.S.S.R., Moscow). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1950, 203-8.—Compds. of the type R_3SnX_3 may be prepd. from the corresponding Pb compds., and in turn are convertible into Tl analogs. Addn., with ice cooling, of 4.57 g. $Pb(OAc)_4$ in $CHCl_3$ to 10 g. Et_4Pb, followed by standing 5 hrs., gave 3.3 g. Pb acetate, m. 200-1°, and 5.5 g. *Et_3PbOAc*, m. 100-00.5° (from the mother liquor). Refluxing 4 g. Ph_4Pb and 5.5 g. $Pb(OAc)_4$ 15 hrs. in dry $CHCl_3$ with a trace of AcOH, followed by filtration of the Pb acetate and evapn., gave 3.7 g. (46%) *$Ph_3Pb(OAc)_3$*, m. 200-1° (from C_6H_6). Refluxing 3 g. Ph_3PbCl_3 and 3 g. Hg in dry Me_2CO 30 hrs., concn., and extn. with hot C_6H_6 gave 16% *$PhHgCl$*, m. 250-1° (from EtOH). Refluxing 9 g. Et_3PbCl_3 and 5 g. $SnCl_2$ in dry EtOH 15 hrs. gave on filtration and evapn., followed by extn. with hot ligroin, 3.3 g. *Et_3SnCl_3*, m. 84° (51%). A similar reaction with Ph_3PbCl_3 in Me_2CO gave in 50 hrs. 13.6% *Ph_3SnCl_3*, m. 42°, while Me_3PbCl_3 (in EtOH soln.) gave 47% *Me_3SnCl_3*, m. 107.5-8.0° (from petr. ether). Dropwise addn. of 1.5 ml. 20% NaOH to 0.3 g. $(p\text{-}MeC_6H_4)_3SnCl_3$ and 0.31 g. $TlCl_3.4H_2O$ in 9 ml. boiling EtOH, followed by refluxing 5 min. and addn. of 5 ml. concd. HCl, gave 62% *$(p\text{-}MeC_6H_4)_3TlCl_3$*, does not m. 300° (from pyridine). Addn. of dry Na_2CO_3 (1.8 g.) to 1 g. $(p\text{-}EtO_2CC_6H_4)_3SnCl_3$ and 0.78 g. $TlCl_3.4H_2O$ in boiling EtOH, followed by treatment with 10 ml. HCl, gave 0.62 g. (36%) *$(p\text{-}EtO_2CC_6H_4)_3TlCl_3$*, does not m. 310° (from pyridine); 20% NaOH instead of Na_2CO_3 gave an impure product. However addn. of 3 ml. 20% NaOH to 0.5 g. $(p\text{-}ClC_6H_4)_3SnCl_3$ and 0.45 g. $TlCl_3.4H_2O$ in hot EtOH gave, after a similar treatment to the above, 86% *$(p\text{-}ClC_6H_4)_3TlCl_3$* darkens at 200° (from aq. pyridine). G. M. Kosolapoff

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C.A.

Addition of iodine trichloride to unsaturated acids. E. M. Bralnina and R. Kh. Freidlina (Acad. Sci. U.S.S.R., Moscow). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1950, 315-20.—Addn. of 2.04 g. ICl_3 in 5 ml. NaCl soln. to 1 g. $(CCO_2H)_2$ and standing 24 hrs. gave, after washing with satd. NaCl, 0.6 g. (20%) *iodosochloride* of *α-chloroacrylic acid*, m. 144° (cf. Thiele and Peter, *Ber.* 38, 2842(1905)); the product, $ClC:CH.ICl.O.CO$, can be crystd. from H_2O. Similarly, $(CCO_2Me)_2$ in 20 days gave 20% of the corresponding $MeO_2CC:CCl.CO.O.ICl$, m. 168-70° (decompn.) (from the crude ppt. by washing with H_2O, EtOH, and ligroin). Fumaric acid similarly gave in 20 days 57% $CH_2.CHCl.CO.O.IOCOCH:CHCO_2H$, a nonfusible solid, sol. in Et_2O, which with pyridine yields a *monopyridine salt*, m. 124.5-5.0° (from EtOH). $CH_2:CHC:CH$ (10.5 g.) and 50 g. iodine in 100 ml. abs. EtOH after 2 days gave 75% of a red oil, b_3 91.5-2.0° (evolution of some iodine). assigned the structure $CH_2:CHCl:CHI$; chlorination of this at −30° in petr. ether-$CHCl_3$ gave a solid, $CH_2:CHCl:CHICl_3$, contg. 18.8% active Cl; the latter (1 g.) with 2 g. $PhSnCl_3$ and 5 ml. 15% HCl at −30° gave overnight 87% Ph_2ICl, decomp. 220°, *picrate*, m. 136-7°. G. M. K.

Chlorination with peroxides as catalysts. IV. Chlorination of methyl formate. Shozo Yura (Kyoto Univ.). *J. Chem. Soc. Japan*, Ind. Chem. Sect., 31, 157-8(1948). In scattered light, chlorination of HCO_2Me stopped with the formation of $ClCO_2CH_2Cl$ (I), a slight amt. of $ClCO_2CHCl_2$ (II) being formed on continued chlorination. In the dark no chlorination occurred, but on addn. of 1% Bz_2O_2 II was obtained, but decompd. on continued chlorination. In the dark completely dry HCO_2Me gave II in the presence of 1% Bz_2O_2 and $ClCO_2CCl_3$ in the presence of Ac_2O_3. K. H.

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10

Reaction of titanium tetrachloride with alcohols in the presence of ammonia and pyridine. O. V. Nogina, R. Kh. Freidlina, and A. N. Nesmeyanov. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* **1950**, 327-30.—$TiCl_4$ does not yield $(RO)_4Ti$ in reactions with ROH unless HCl is eliminated immediately by means of alk. reagents. Slow addn. of 53 g. $TiCl_4$ to 50 g. EtOH, 85 g. pyridine, and 200 ml. C_6H_6 with ice cooling, followed by 2 hrs. at 60-70° and filtration, gave 67% $(EtO)_3TiCl$, b_{10} 147.5-50.0°; possibly the pyridine-HCl remaining in the filtrate reacted with the neutral ester during distn. $(EtO)_4Ti$ (15.5 g.), 8 g. pyridine-HCl, and 95 ml. C_6H_6 heated 2 hrs. at 70-5° gave 77% $(EtO)_2TiCl$; $(BuO)_4Ti$ does not react in these conditions. Addn., with ice cooling, of 35 g. $TiCl_4$ to 200 g. BuOH, followed by passage of NH_3 to a 13.5-g. wt. gain, and heating 2 hrs. at 70°, gave after filtration 48% $(BuO)_4Ti$, b_{10} 100-2°; a 60% yield is obtained with 120 g. BuOH in 250 ml. C_6H_6 treated with 60 g. $TiCl_4$, followed by NH_3 as above; reaction in C_6H_6 with pyridine as the acid-binding agent gave a 54% yield. Slow addn. of 25 g. $TiCl_4$ to 150 g. CH_2CHCH_2OH with cooling, followed by dry NH_3 (wt. gain 9.5 g.) and heating 1 hr. at 70-80° gave 38% $(CH_2CHCH_2O)_4Ti$, b_{10} 141-2°; a 42% yield is obtained when C_6H_6 is used as a diluent; the product $b_{0.5}$ 156-8°. Slow addn. of 25 g. $TiCl_4$ to 150 g. EtOH with ice cooling, followed by dry NH_3 (wt. gain 9.4 g.), then heating 40 min. at 70°, filtration on cooling, concn. *in vacuo*, dilu. with C_6H_6 to ppt. residual halide, and repeated filtration, gave 70% $(EtO)_4Ti$, b_{10} 128-9°. Addn. of 16.0 g. $TiCl_4$ to 35 g. cyclohexanol in 150 ml. C_6H_6 with cooling, followed by dry NH_3 for 10 hrs. with cooling, filtration, and distn. gave 53% $(C_6H_{11}O)_4Ti$, $b_{0.5}$ 204-5°, b_{10} 190.5-2.0°. G. M. Kosolapoff

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A

Action of magnesium on *N*-(2-bromoethyl)-*N*-methyl **aniline.** A. N. Nesmeyanov, R. Kh. Freidlina, and F. I. Kan. *Izvest. Akad. Nauk S.S.S.R., Otdel Khim. Nauk* 1950, 31-6.—Reaction of 3 g. Mg and 27 g. $BrCH_2CH_2NMePh$ in 60 ml. Et_2O in a N atm., completed by 1 hr. on a steam bath, gave 38% *PhNHMe* (*picrate*, m. 143-5°), 30% C_2H_4 (isolated as the dibromide), and 4.8 g. (*PhMeNCH₂CH₂*)₂, whose *dipicrate* m. 202-3° (from Me_2CO). The product from 14.6 g. $BrCH_2CH_2NMePh$ and 1.6 g. Mg in Et_2O with 6.2 g. PrCHO gave, upon aq. treatment, a *yellow product*, whose *picrate* m. 124-5°; the same product, along with some PhNHMe, was obtained by addn. of PrMgBr to an equimolar amt. of PhNMeH. Hence, Gilman and Heck (C. A. 23, 4188), Braun, *et al.* (C. 12, 2657), erred in claiming the formation of $PhNMeCH_2CH_2MgX$; the latter is extremely unstable and immediately cleaves to C_2H_4 and PhMeNMgX. The picrate, m. 124-5°, has a N content corresponding to the picrate of $MePhNCH_2CH_2CHOHPr$, but its C and H analyses (unstated) show that it is not this compd.

G. M. Kosolapoff

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FREYDLINA, R. Kh., KURSANOV, D.N., KABACHNIK, M.I., KAVERZNEVA, Ye.D., PRILEZHAYEVA, Ye.N. and SOKOLOV, N.D.

"The Current State of Chemical Structure, Usp.Khim., 19, No.5, pp 529-544, 1950.

Translation W-16104, 30 Dec 50

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R.Kh.; NOGINA, O.V.; NESMEYANOV, A.N.

Bromine

Effect of bromine on alkylchloracarbonates. Uch. zap. Mosk. un., No. 132, 1950.

9. Monthly List of Russian Accessions, Library of Congress, October 1952, Uncl.

APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3"

"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

NESMEYANOV, A.N.: FREYDLINA, R.Kh.: KOCHETKOV, A.K.

Dimethylvinyl Carbinol Acetate

1, 6-Addition of mesitylmagnesium bromide to dimethyl vinylcarbinol acetate. Uch. zap. Mosk. un., No. 132, 1950.

9. Monthly List of Russian Accessions, Library of Congress, October 1952, Uncl.

APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3"

"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

CA. FREYDLINA, R.Kh.

Addition of organomagnesium compounds to 1,1-dimethylallyl acetate. A. N. Nesmeyanov, R. Kh. Friedlina, and A. K. Kochetkov. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1951 273-9; cf. C.A. 44, 3020e.—EtMgBr and PhMgBr react with $CH_2:CHCMe_2OAc$ (I), by the way of 1,2- and 1,6-addns. 1,3,5-$Me_3C_6H_2MgBr$ (II) adds in the 1,4-manner to $Me_2C:CHCH_2OAc$ (III). All facts about addn. of RMgX to allyl esters are explainable without a dissociative mechanism, but on assumption that the single link between the 3rd and 4th C atoms can conjugate with the CO and C:C links, so that 2nd, 4th, and 6th C atoms are subject to the attack by nucleophilic reagent, and by steric effects one can direct the point of reaction to the desired atom. EtMgBr (from 30 g. EtBr) in Et_2O added slowly to 30 g. I in Et_2O and refluxed 5 hrs. gave, after removal of MeCOEt as a semicarbazone, 33.5% *2-methyl-2-hexene*, b_{760} 94-4.5°, n_D^{20} 1.4077, d_4^{20} 0.7091 (ozonolysis gave Me_2CO and $PrCO_2H$). Similarly PhMgBr gave AcPh and 25% *2-methyl-4-phenyl-2-butene*, isolated as the *dibromide*, m. 63.5-4.0°. II (from 35 g. RBr) and 17 g. III gave 8.7 g. mesitylene and 4.6 g. liquid, b_{12} 142.5-4.0°, n_D^{20} 1.5196, d_4^{20} 0.9042, identified as *2-methyl-4-mesityl-2-butene*, yielding the *2,3-dibromide*, m. 71-1.5°.

G. M. Kosolapoff

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

USSR/Chemistry - Halogenated Hydrocarbons Sep/Oct 51

"Synthesis and Properties of Certain Polyhalogen Derivatives of Propane and Propene Containing the Trichloromethyl Group," A. N. Nesmeyanov, R. Kh. Freydlina, V. I. Firstov, Inst of Org Chem, Acad Sci USSR

"Iz Ak Nauk SSSR, Otdel Khim Nauk" No 5, pp 505-511

Action of KOH on $CCl_3CH_2CH_2Cl$ yielded $CCl_2=CHCH_2Cl$ and $CCl_3CH=CH_2$ (latter compd never before prepd). By series of reactions starting

195T5

USSR/Chemistry - Halogenated Hydrocarbons (Contd) Sep/Oct 51

from these 2 compds, prepd 8 new compds contg CCl_2 and CCl_3 groups. Of compds contg CCl_3 group, 2 were Br-substituted.

195T5

FREYDLINA, R. Kh.

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R. Kh.

Sep/Oct 51

USSR/Chemistry - Oxime Ethers

"Synthesis of o-Allyl Ethers of Oximes," A. N. Nesmeyanov, A. K. Kochetkov, R. Kh. Freydlina, Inst of Org Chem, Acad Sci USSR

"Iz Ak Nauk SSSR, Otdel Khim Nauk" No 5, pp 512-517

Interacted allylbromide (I) with ald- and ket-oximes in presence of Na ethylate to obtain good yield of o-allyl oxime ethers, easily purified by distn, but no N-allyl derivs under conditions in question. Action of I on KON $(SO_3K)_2$ gave 60% yield of o-allylhydroxylamine (II), b 97.5-99° C. Reacted II with oximes to prep a number of o-allyl oxime ethers. In all, prepd 6 heretofore unknown o-allyl oxime ethers.

PA 195T6

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

USSR/Chemistry - Organic Titanium Compounds

Sep/Oct 51

"Action of Chlorine and Bromine on Alkylorthotitanates," A. N. Nesmeyanov, R. Kh. Freydlina, O. V. Nogina, Inst of Org Chem, Acad Sci USSR

"Iz Ak Nauk SSSR, Otdel Khim Nauk" No 5, pp 518-523

Found that action of Cl_2 and Br_2 on Et, n-Pr, and Bu orthotitanates gives good yield of compds of type $(RO)_2TiHal_2 \cdot ROH$ and that orthoalkyltitanates cause ester condensation of aldehydes according to Tishchenko. Synthesized following previously unknown compds: $(RO)_2 TiBr_2 \cdot ROH$ (R = Et, Pr, Bu, resp);

195T7

USSR/Chemistry - Organic Titanium Compounds (Contd)

Sep/Oct 51

$(RO)_2TiCl_2 \cdot ROH$ (R = Pr, Bu, resp); $(PrO)_3TiCl$ by interaction of pyridine chlorohydrate with Pr orthotitanate; and $(RO)_4Ti$ (R = ClEt, Bz, MeOEt, EtOEt, resp by re-esterification.

195T7

FREYDLINA, R. Kh.

APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3"

"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R.KH.

Chem Abs V48
1-25-54
Organic chemistry

(CA 48 no.2: 567 '54)

Preparation of alkoxytitanium trichlorides from titanium tetrachloride and alcohols. A. N. Nesmeyanov, R. Kh. Freidlina, and O. V. Nogina. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1953, 1037–40; cf. Jennings, *et al.*, *C.A.* 30, 5180[4].—Reaction of $TiCl_4$ with small proportions of ROH yields $ROTiCl_3$ as the sole product. The reagents thoroughly dry and the reaction must be protected against atm. moisture. To 142.5 g. $TiCl_4$ and 800 ml. petr. ether (b.50–80°) was gradually added 30 g. PrOH in 200 ml. petr. ether, the mixt. was refluxed 3 hrs., concd., and chilled, yielding a ppt. which was sepd. and distd. giving after 2 distns. 96.5% *$PrOTiCl_3$*, b_{11} 83–5°, b_{20} 100–2°, m. 65–6°. Similarly 142 g. $TiCl_4$ and 23 g. EtOH in petr. ether gave, after 8 hrs. reflux, decantation from a ppt. concn. of the soln., and chilling, 87.5% *$EtOTiCl_3$*, b. 185–6°. $TiCl_4$ (95 g.) and 24.7 g. iso-BuOH in 225 ml. CCl_4 gave after 9 hrs. reflux and chilling, nearly 100% *iso-$BuOTiCl_3$*, b_9 92–4°, m. 81–3°. iso-AmOH (44 g.) and 142.5 g. $TiCl_4$ in petr. ether gave after 5 hrs. reflux 95.5% *iso-$AmOTiCl_3$*, b_{11} 110–11°, m. 50–60°; C_3H_7OH and $TiCl_4$ similarly gave after 3 hrs. reflux nearly 100% *$C_3H_7OTiCl_3$*, b_8 120–2°, m. 47.5–9.0°; $MeOCH_2CH_2OH$ (38 g.) and 142.5 g. $TiCl_4$ in 275 ml. CCl_4 gave after 4 hrs. reflux nearly 100% *$MeOCH_2CH_2OTiCl_3$*, which sublimed slowly at 160–80° at 5 mm., and the sublimed product, m. 164–6°; it was insol. in petr. ether, CCl_4, C_6H_6, or Et_2O. G. M. Kosolapoff

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

USSR/Chemistry - Titanium Compounds Jan/Feb 52

"A New Method for Synthesizing Trialkoxytitanium Chlorides," O. V. Nogina, R. Kh. Freydlina, A. N. Nesmeyanov; Inst of Org Chem, Acad Sci USSR

"Iz Ak Nauk, Otdel Khim Nauk" No 1, pp 74-77

The new synthesis is based on the re-etherification of thiethoxy titanium chloride, and proceeds as follows:

$$(C_2H_5O)_3TiCl + 3\ ROH \leftrightarrows (RO)_3TiCl + 3\ C_2H_5OH.$$

208T6

USSR/Chemistry - Titanium Compounds (Contd) Jan/Feb 52

A number of heretofore unknown compds was synthesized by this method, which is quick and gives an almost quant yield.

208T6

FREYDLINA, R. KH.

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FREYDLINA, R. KH.

Chem 5

Chem Abs v48
1-25-54
Organic Chemistry

1952

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Allyl orthotitanate. O. V. Nogina and R. Kh. Freidlina. Akad. Nauk S.S.S.R., Inst. Org. Khim., Sintezy Org. Soedinenii, Sbornik 2, 10-11; cf. C.A. 44, 8854i.—In app. provided with drying tubes for exclusion of moisture 102 g. dry allyl alc. and 250 ml. dry C_6H_6 is treated with ice cooling with 60 g. $TiCl_4$ over 30-40 min., after which the mixt. is satd. with NH_3 (not less than 25 g. gain in wt.) and filtered. The filtrate yields 41-6% $(CH_2:CHCH_2O)_4Ti$, b_1 141-2°, which is very hygroscopic and readily hydrolyzed.

G. M. Kosolapoff

1-28-54

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FREYDLINA, R. KH.

5 Chem
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Chem Abs V48
1-25-54
Organic Chemistry

1,1,1-Trichloropropene. R. Kh. Freidlina and V. I. Firsov. Akad. Nauk S.S.S.R., Inst. Org. Khim., Sintezy Org. Soedinenii, Sbornik 2, 134-5(1952).—To 50 g. $CCl_3CH_2CH_2Cl$ is added with stirring and cooling to 0° over 6 hrs. 20 g. KOH in 100 ml. abs. EtOH. Dilg. with H_2O, drying the org. layer, and then distg. gave 50% $CCl_3CH{:}CH_2$ (I), b. 101-3° (pure b. 101-2°, n_D^{20} 1.4680, d_{20} 1.3292), along with some 3% *1,1,3-trichloro-1-propene*, b. 131-3°. I heated in steel ampul 3 hrs. at 150° isomerizes to the latter substance, b. 131-2°, n_D^{20} 1.4960, d_{20} 1.3940. G. M. K.

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FREYDLINA, R. Kh.

USSR/Chemistry - Titanium Organic Compounds 21 Jul 52

"Investigation of the Hydrolysis and Polycondensation of Ethyl Orthotitanate," Acad A.N. Nesmeyanov, E.M. Braynina, R. Kh. Freydlina, Inst of Org Chem, Acad Sci USSR

"Dok Ak Nauk SSSR" Vol 85, No 3, pp 571-573

Ethyl orthotitanate reacts with water to form $(C_2H_5O)_8Ti_3O_2$, a polymer with the structure $(C_2H_5O)_3Ti - O - Ti(OC_2H_5)_2 - O - Ti(OC_2H_5)_3$.

(CA 47 no. 15: 7433 '53)

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APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3"

"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

USSR

Synthesis of α,α,α-trichloro-ω-aminoalkanes and α,α-dichloro-ω-aminoalkenes. A. N. Nesmeyanov, L. I. Zakharkin, and R. Kh. Freidlina (Inst. Org. Chem., Acad. Sci. U.S.S.R., Moscow). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1954, 34–40.—Heating 25 g. $Cl(CH_2)_3CH{:}CCl_2$ (I) with 100 ml. MeOH contg. 12 g. NH_3 in a steel ampul 8 hrs. at 80-5° gave 73% $H_2N(CH_2)_3CH{:}CCl_2$, b_7 68-9°, n_D^{20} 1.4899, d_{20} 1.1736; *N-Bz deriv.*, m. 55-6° (from ligroine). I (62 g.) refluxed with 32 g. KCN in 55 ml. H_2O and 150 ml. EtOH with 3 g. NaI 10 hrs. gave 72% $NC(CH_2)_3CH{:}CCl_2$, b_7 80-1°, n_D^{20} 1.4815, d_{20} 1.2018. This (35 g.) and 35 g. $Cl(CH_2)_3CCl_3$ hydrogenated 3 hrs. in 100 ml. MeOH over 5 g. Raney Ni at 75° and 100 atm. H yielded 22% $H_2N(CH_2)_4CH{:}CCl_2$, b_3 83-4°, n_D^{20} 1.4865, d_{20} 1.1331. Hydrolysis of this with H_2SO_4 gave $H_2N(CH_2)_5CO_2H$, m. 200-1°; *N-Bz deriv.* of the *amine*, m. 36.5-7.5° (from ligroine). Heating 12 g. $Cl(CH_2)_3CH{:}CCl_2$ with 8 g. NH_3 in 60 ml. MeOH 8 hrs. at 80-5° gave 56.2% $H_2N(CH_2)_3CH{:}CCl_2$, b_7 102-3°, n_D^{20} 1.4842, d_{20} 1.1039; *N-Bz deriv.*, m. 37.5-8° (from ligroine). Similarly $Cl(CH_2)_3CH{:}CCl_3$ gave 50.5% $H_2N(CH_2)_3CH{:}CCl_3$, b_3 100-1°, n_D^{20} 1.4822, d_{20} 1.0599; *N-Bz deriv.*, m. 44.5-5.5° (from ligroine). Heating 25 g. $Cl(CH_2)_4CCl_3$ with 20 g. NH_3 in 100 ml. MeOH 10 hrs. at 85-90° gave 58.2% $H_2N(CH_2)_5CCl_3$, $b_{1.5}$ 69-70°, n_D^{20} 1.4862, d_{20} 1.2019; *N-Bz deriv.*, m. 95-6° (from dil. EtOH). Hydrolysis with concd. H_2SO_4 gave $H_2N(CH_2)_6CO_2H$, m. 165-6°. On standing $H_2N(CH_2)_5CCl_3$ deposits a crvst. *HCl salt*, m. 229-30° (from H_2O); heated above 130-40°, the amine undergoes a

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

USSR.

Some reactions of 1,1,1-trichloropropylene and 1,1,3-trichloropropylene. A. N. Nesmeyanov, [illegible] Zakharkin, V. N. Kost, and R. Kh. Freidlina (N. D. Zelinskii Inst. Org. Chem., Acad. Sci. U.S.S.R., Moscow). *Izvest. Akad. Nauk S.S.S.R., Otd. Khim. Nauk* 1954, 258-65.—$CCl_3CH:CH_2$ (I) (10 g.) heated with 10 g. Et_2NH in MeOH 8 hrs. at reflux gave 9.3 g. $CCl_2:CHCH_2NEt_2$, b_1 65-6°, n_D^{20} 1.4708, d_4 1.0693; *HCl salt*, m. 139-40°. Similar reaction of $CCl_2:CHCH_2Cl$ (II) gave 65% of the same substance. II (30 g.) added to the mixt. of 3.2 g. $CH_2(CO_2Et)_2$ and 4.6 g. Na in abs. EtOH gave after 3 hrs. refluxing 50% $CCl_2:CHCH_2CH(CO_2Et)_2$ (III), $b_{0.4}$ 102-3°, n_D^{20} 1.4633, d_4 1.2135, and 11.2 g. $(CCl_2:CHCH_2)_2C(CO_2Et)_2$, (IV), $b_{0.4}$ 138-40°, m. 39-40°. Similar reaction of 21 g. I with 24 g. $CH_2(CO_2Et)_2$, 3.5 g. Na, and 50 ml. EtOH gave after 5 hrs. 10.3 g. III and 10.6 g. IV. I refluxed with aq. alc. Na_2S 4 hrs. gave 62% $(CCl_2:CHCH_2)_2S$, b_3 101-2°, n_D^{20} 1.5028, d_4 1.4450, which with 30% H_2O_2 in AcOH gave the *sulfone*, m. 113-14°. Heating 15 g. I with 5 g. Na in 50 ml. abs. MeOH 3 hrs. gave 50% $MeOCH_2CH:CCl_2$, b. 132-3°, n_D^{20} 1.4558, d_4 1.2117. Similar reaction of II was complete in 1 hr. and gave the same product as above, b. 131-2°; this with Cl in the dark at 60° gave 61% $C_4H_6Cl_4O$, $b_{0.4}$ 97-8°, n_D^{20} 1.5093, d_4 1.5740, the formation of which involved both addn. and substitution with loss of HCl. Heat-

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Nesmeyanov, A.N.

ing 50 g. I with 32 g. $(CuCN)_2$ to 130°, then to 160° until refluxing stopped, gave 30 g. *dichlorobutenonitrile* (V), b_{11} 77–8°, n_D^{20} 1.4831, d_{20} 1.3142; similar reaction of II gave a lower yield of the same product; treatment of the product with Cl gave *tetrachlorobutanonitrile*, m. 44°. Only a slight yield of the dichloronitrile formed from II and KCN in aq. alc. soln. V (2 g.) heated with 10 ml. 90% H_2SO_4 to 100–20° gave 0.7 g. $(CH_2CO_2H)_2$, indicating that V is $CCl_2:CHCH_2CN$. V treated with Cl in refluxing CCl_4 with illumination gave 94% $CCl_3CHClCH_2CN$, $b_{0.5}$ 105–7°, m. 43.5–4.0° (from petr. ether). This (19 g.) in 25 ml. MeOH was added to cooled 5 g. KOH in 50 ml. MeOH; after 24 hrs. at room temp. there was obtained 12.5 g. $CCl_2CH:CHCN$, b_7 68–9°, n_D^{20} 1.5082, d_{20} 1.4237; with excess KOH at higher temp. there is formed $CCl_2CH(OMe)CH_2CN$, $b_{0.5}$ 99.5°, n_D^{20} 1.4820, d_{20} 1.3879. To 21 g. I was added BuMgBr from 17 g. BuBr; after refluxing 10 min. and treatment with H_2O there was obtained 68% *1,1-dichloroheptene*, b_8 59–60°, n_D^{20} 1.4585, d_{20} 1.0427, which hydrolyzed to enanthic acid; anilide, m. 64–5°. BuMgBr and II gave 31% same product. Addn. of 10 g. I to 6 g. Raney Ni in EtOH caused a vigorous reaction; after 20 min. refluxing the mixt. yielded 48% $CCl_2:CHCH_2CH_2CH:CCl_2$, b_6 98–100°, n_D^{20} 1.5149, d_{20} 1.3628, which hydrolyzed to adipic acid. I (15 g.) added to a mixt. of 20 g. $CCl_2:CH_2$ and 1 g. $AlCl_3$ at 15–20°, stirred 15 min. and poured into H_2O, gave 76% *1,1,5,5,5-pentachloro-1-pentene*, b_{10} 101–4°, n_D^{20} 1.5125, d_{20} 1.4707 which hydrolyzed with H_2SO_4 to glutaric acid. Similarly I and $CHCl:CCl_2$ gave *1,1,4,5,5,5-hexachloro-1-pentene*, b_{10} 110–20°, n_D^{20} 1.5291, d_{20} 1.5817; similarly $CCl_2:CH_2$ and I gave *1,1,4,5,5-pentachloro-1-pentene*, b_7 105–7°, n_D^{20} 1.5225, d_{20} 1.5077.

G. M. Kosolapoff

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FREYDLINA, R. Kh.

USSR/ Chemistry Organic chemistry

Card : 1/1 Pub. 40 - 4/27

Authors : Nesmeyanov, A. N., Zakharkin, L. I., Kost, V. N., and Freydlina, R. Kh.

Title : Allyl regrouping in polychlorobutenic acids and about certain errors made by Auwers and Wissebach in their work regarding allyl regrouping

Periodical : Izv. AN SSSR. Otd. khim. nauk 4, 604 - 609, July - August 1954

Abstract : The relation of $CHCl_2 - CH = CH - CO_2H$ and $CCL_2 = CH - CH_2CO_2H$ acids and the corresponding relation of their derivatives to prototropic allyl regrouping, was investigated. The errors made by two German chemists, K. A. Auwers and H. Wissebach, in two-stage reduction of gamma,gamma, gammatrichlorocrotonic acid into plain crotonic acid through double isomerization, are pointed out. Seven references: 3 USSR; 2 USA and 2 German (1923 - 1954).

Institution : Acad. of Sc. USSR, Institute of Organic Chemistry

Submitted : August 23, 1953

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FREYDLINA, R. Kh.

USSR/Chemistry - Organic chemistry

Card 1/2 Pub. 40 - 6/27

Authors : Nesmeyanov, A. N.; Freydlina, R. Kh.; and Braynina, E. M.

Title : Reaction of alkylorthetitanates and titanium tetrachloride in a piperidine medium

Periodical : Izv. AN SSSR. Otd. khim. nauk 6, 987-991 - 1954

Abstract : Experiments showed that alkylorthetitanates, alkoxytitanium chlorides, di- and trichlorides together with piperidine form certain crystalline binary compounds of the $(RO)_n TiCl_{4-n} \cdot C_5H_{10}NH$ composition. It is shown that the reaction, which is inversely disproportional between the alkylorthotitanates and titanium tetrachloride, passes smoothly in the presence of a estimated amount of piperidine yielding large quantities of

Institution : Acad. of Sc., USSR, The N. D. Zelinskiy Institute of Organic Chemistry

Submitted : January 19, 1954

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Periodical : Izv. AN SSSR. Otd. khim 6, 987-991, Nov-Dec 1954

Card 2/2 Pub. 40 - 6/27

Abstract : binary piperidine-alkoxytitanium chloride compounds. The chemical composition of the binary compounds is described. Three references: 2 USSR and 1 USA (1950-1954).

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FREIDLINA, R. KH.

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Reaction of reverse disproportionation in the series of organic compounds of titanium. A. N. Nesmeyanov, B. M. Braynina, and R. Kh. Freidlina. *Doklady Akad. Nauk S.S.S.R.* 94, 243-52(1954); cf. *C.A.* 46, 7038i; 48, 567e; Jennings, *et al.*, *C.A.* 30, 5180[1].—Examples of "reverse" disproportionation between $TiCl_4$ and alkoxy derivs. of Ti are described. Thus 2.02 g. $TiCl_4$ added to 7.31 g. $Ti(OEt)_4$ in 10 ml. C_6H_6 with ice cooling gave after a few min. 86% $(EtO)_3TiCl$, b_{10} 177-7.5°, b_5 136-7°. Similarly 0.02 mole $(EtO)_4Ti$ and 0.02 mole $TiCl_4$ mixed in petr. ether-C_6H_6 with cooling gave 79% $(EtO)_2TiCl_2$, m. 40-50° (with darkening) (from C_6H_6). $(EtO)_4Ti$ (0.01 mole) with 0.033 mole $TiCl_4$ in petr. ether-C_6H_6 similarly gave 84% $EtOTiCl_3$, m. 81-2°. $(PrO)_4Ti$ (0.03 mole) and 0.01 mole $TiCl_4$ in petr. ether-C_6H_6 let stand overnight gave 79% $(PrO)_3TiCl$, b_1 136-7°. Similarly 0.01 mole $(BuO)_4Ti$ and 0.03 mole $TiCl_4$ gave 62% $BuOTiCl_3$, m. 45-55° (from C_6H_6). $(BuO)_3TiCl$ (0.02 mole) with 0.04 mole $TiCl_4$ gave 53% $BuOTiCl_3$. Thermal decompn. or disproportionation of the above halides during distn. depends on the method of prepn. Thus, $(PrO)_3TiCl_3$ prepd. from PrOH and $TiCl_4$ can be distd. readily (159°/18 mm.), but the same product prepd. from AcCl and $(PrO)_4TiCl$ suffers much decompn. on distn. The compds. of the general type $(RO)_nTiCl_{4-n}$, cited above, can be usually isolated rapidly in the form of adducts with 1 mole piperidine, which can be purified by crystn. from petr. ether-C_6H_6 but are v ry hygroscopic.

G. M. Kosolapoff

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Trialkoxytitaniums and dialkoxytitanium oxides. A. N. Nesmeyanov, O. V. Nogina, and B. Kh. Freidlina (Sz. D. Zelinskii Inst. Org. Chem., Acad. Sci. U.S.S.R., Moscow). Doklady Akad. Nauk S.S.S.R. 95, 813-15(1954).—To 1.7 g. K was added 3 g. $(PrO)_3Ti$ in 30 ml. PrOH; the resulting ppt. was sepd., washed with PrOH and petr. ether, and dried in N under low pressure at room temp.; the product was a pink-lilac solid, $(PrO)_3Ti$; brief drying left brown $(PrO)_3Ti$.-2PrOH. Pink-lilac $(BuO)_3Ti$ was similarly obtained. Contact with air yielded BuOH or PrOH and a yellowish powdery $(PrO)_3TiO$, m. 100-13°, or $(BuO)_3TiO$, m. 112-15°. These are sol. in org. solvents in contrast to $(RO)_3Ti$ derivs. The oxidation also yields some corresponding aldehyde besides the ROH and is believed to be a free-radical reaction with $(RO)_3TiO$-intermediate.

G. M. Kosolapoff

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13778* (Chemical Transformations of Trichloromethyl Group in Saturated Polychlorohydrocarbons.) Khimicheskie prevrashcheniia trikhlorometil'noi gruppy v predel'nykh polikhlorugleverodakh. A. N. Nesmeianov, [illegible] Kh. Freidlina, and L. I. Zakharkin. Doklady Akademii Nauk SSSR, v. 96, no. 1, May 1, 1954, p. 87-90.

Formulations of regularities in compounds. 10 ref.

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R.KH.

USSR/ Chemistry

Card : 1/1

Authors : Nesmeyanov, A. N., Academician; Freydlina, R. Kh., and Zakhrakin, L. I.

Title : Conversion of 1,1,1,3-tetrachloro-3-alkoxypropanes into esters of beta, beta-dialkoxypropionic acid (propionate).

Periodical : Dokl. AN SSSR, 97, Ed. 1, 91 - 94, July 1954

Abstract : The reaction leading to the conversion of tetrachloro-alkoxypropanes into propionates (esters of β,β-dialkoxypropionic acid) under the effect of energetic alcohols, is described. The problem of whether the chlor-atoms in the trichloromethyl group are directly displaced by alkoxy groups is discussed. Data regarding the experiment and instruments used are included. Six USA references.

Institution : Acad. of Sc. USSR, Institute of Elementary-Organic Compounds

Submitted : May 12, 1954

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

USSR/Chemistry - Allyl regrouping

Card 1/1 Pub. 22 - 30/56

Authors : Nesmeyanov, A. N., Academician; Freydlina, R. KH.; and Zakharkin, L. I.

Title : Regrouping of 1,1-dichloro-3-alkoxy-3arylpropenes-1 into beta-arylacrylic acids

Periodical : Dok. AN SSSR 99/5, 781-783, Dec 11, 1954

Abstract : Another example of allyl regrouping in a dichloropropene system is presented. The conversion of 1,1-dichloro-3-alkoxy (oxy)-3-arylpropenes-1 into beta-arylacrylic acids, by heating the propenes in an acetic acid solution in the presence of hydrochloric acid, is described. The physico-chemical properties of the regrouping products (acids) are listed. Eleven references: 3-USA: 5-USSR; 2-French and 1-Italian (1881-1954).

Institution : Academy of Sciences USSR, Institute of Elementary Organic Compounds

Submitted : October 7, 1954

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FREYDLINA, R. Kh

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Chem

Synthesis of alkoxytitanium bromides and alkoxypolytitanoxane chlorides. A. N. Nesmeyanov, E. M. Brainina, and R. Kh. Freidlina (Inst. Hetero-org. Compds., Moscow). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1955, 838-43; cf. *C.A.* 47, 7433b; Boyd, *C.A.* 48, 9250g.—All expts. described below were performed with rigorous exclusion of H_2O. $(EtO)_4Ti$ (33.7 g.) in 15 ml. abs. EtOH slowly treated with 1.31 g. H_2O in 10 ml. abs. EtOH, refluxed briefly, filtered hot, and cooled, yielded 75% $(EtO)_3Ti_2O_3$ (I), m. 78-85° (from EtOH); no other polymer was found in the residual soln. I (10.7 g.) heated 2 hrs. with 20 ml. BuOH, freed of ROH by distn., heated again with fresh BuOH, and the ROH evapd. gave a viscous liquid which could not be [illegible]d.; heated *in vacuo* to 90°, the product was identified as $(BuO)_3Ti_2O_3$, a very hygroscopic and readily hydrolyzable viscous mass. Heating to 220° gave a distillate of $(EtO)_4Ti$, leaving behind a solid polymer. Similar transesterification with $C_6H_{13}OH$ gave a liquid $(C_6H_{13}O)_3Ti_2O_3$ (II), which on heating to 250-60° yielded $(C_6H_{13}O)_4Ti$, b_2 199°, n_D^{20} 1.4840. I (5.36 g.) in 5 ml. petr. ether treated with cooling with 0.78 g. AcCl in 3 ml. petr. ether, then heated 2 hrs. at 70-5°, cooled, filtered, and distd., yielded EtOAc and a solid residue identified after drying *in vacuo* as $(EtO)_3Ti_2O_3Cl$, having an indefinite m.p. and sol. in petr. ether, but could not

1/2

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Synthesis of alkoxytitanium ...

be recrystd. Similarly, 4.8 g. I and 1.4 g. AcCl gave a solid $(EtO)_2Ti_2O_3Cl_2$ (from petr. ether). $(BuO)_4Ti_2O_3$ (11.45 g.) and 2.36 g. AcCl in petr. ether-C_6H_6 gave 92% viscous $(BuO)_6Ti_2O_3Cl_2$, which, heated *in vacuo*, yielded, from 4.33 g. starting material, 1.83 g. $(BuO)_3TiCl$, $b_{2.4}$ 156.5°, n_D^{20} 1.5192, and a solid polymer. II (14.82 g.) and 2.36 g. AcCl similarly gave liquid $(C_6H_{13}O)_6Ti_2O_3Cl_2$, yielding on distn. a polymer and $(C_6H_{13}O)_3TiCl$, b_2 198-201°, n_D^{20} 1.5060. $(EtO)_4Ti$ (1.14 g.) in 6 ml. C_6H_6-petr. ether with 1.84 g. $TiBr_4$ in 5 ml. C_6H_6 gave after prolonged cooling to −10° 79% $(EtO)_3TiBr_3$, m. 47-50°. Similarly, 2.28 g. $(EtO)_4Ti$ and 11.02 g. $TiBr_4$ gave 45% $EtOTiBr_3$, a solid with indefinite m.p. (from C_6H_6-petr. ether). $(EtO)_4Ti$ (2.28 g.) in 3 ml. petr. ether treated with cooling with 1.7 g. piperidine in C_6H_6, then dropwise with 3.67 g. $TiBr_4$ in C_6H_6, and kept overnight, yielded 80% $(EtO)_3TiBr_3.(CH_2)_5NH$. Similarly 0.84 g. $(EtO)_4Ti$, 3.4 g. piperidine, and 3.67 g. $TiBr_4$ gave 75% $(EtO)_3TiBr.(CH_2)_5NH$; the products can be crystd. from C_6H_6-petr. ether. G. M. Kosolapoff

2/2

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FREYDLINA, R. Kh

Condensation of 1,1,1-trichloropropene with aromatic compounds. A. N. Nesmeyanov, R. Kh. Freidlina, and N. A. Semenov (Inst. Heteroorg. Compds., Acad. Sci. U.S.S.R., Moscow). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1955, 993-8; cf. *C.A.* 46, 7031*b*.—To 45 g. $CCl_3CH:CH_2$ (I) and 196 g. PhBr was added 1 g. $AlCl_3$; the vigorous reaction was complete in 5-10 min. and after cooling and aq. treatment, the mixt. gave 76% *3-(p-bromophenyl)-1,1-dichloro-1-propene* (II), b_4 117.5-8°, n_D^{20} 1.5830, d_{20} 1.5532; this (20 g.) and 50 g. 93% H_2SO_4 after 1 hr. at 120° was quenched in H_2O yielding 93% *p*-bromohydrocinnamic acid, m. 135°; with concd. H_2SO_4 at 145° this gave 63% 6-bromohydrindone, m. 111-12°. II (40 g.) chlorinated 6 hrs. at 60° (wt. gain 9.6 g.) yielded in cooling a ppt. identified as 65% *3-(p-bromophenyl)-1,1,1,2-tetrachloropropane*, m. 89°. Reaction of 45 g. I with 180 g. PhCl, and 1 g. $AlCl_3$, as above, gave 74.5% *3-(p-chlorophenyl)-1,1-dichloro-1-propene* (III), b_4 115-16°, n_D^{20} 1.5630, d_{20} 1.3208; this hydrolyzed as above to 82% *p*-chlorohydrocinnamic acid, m. 123°, which with H_2SO_4 at 180° gave 6-chlorohydrindone, m. 79-80°. Chlorination of III at 30° gave 78% *3-(p-chlorophenyl)-1,1,1,2-tetrachloropropane*, m. 90°. I (44 g.) and 93 g. MeOPh with 2 g. $AlCl_3$ heated 3 hrs. at 90° with gradual addn. of 5 g. $AlCl_3$ gave 74% *3-(p-methoxyphenyl)-1,1-dichloro-1-propene* (IV), b_4 118-19°, n_D^{20} 1.5480, d_{20} 1.2307; oxidation with $KMnO_4$ gave *p*-$MeOC_6H_4CO_2H$, m. 184.5°. Heating 66 g. I, 220 g. PhOH, and 4 g. $AlCl_3$ 4 hrs. on a steam bath, dilg. with H_2O and extg. with $CHCl_3$ gave 50% *3-(o-hydroxyphenyl)-1,1-dichloro-1-propene* (V), b_3 116-17°, n_D^{20} 1.5727, d_{20} 1.3050, and 22% *p-isomer*, b_4 130-1°, n_D^{20} 1.5732, d_{20} 1.3057, m. 40.5-1° (after long storage); if $AlCl_3$ be omitted, the same products form in 50% total yield. Methylation of the above *o*-isomer with Me_2SO_4 gave *3-(o-*

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1/2

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7

Nesmeyanov, A.N.

methoxyphenyl)-1,1-dichloro-1-propene, $b_{0.4}$ 109°, n_D^{19} 1.5525, d_4 1.2972; methylation of the *p*-isomer gave IV. Heating 1 mol. PhOH, 1 mole NaOH, and 0.25 mole I in 100 ml. EtOH 6 hrs. at 108° gave 12 g. 3 *phenoxy-1,1-dichloro-1-propene*, b_1 183-4°, n_D^{23} 1.5534, d_4 1.2718, and 16 g. mixed *o*-*p*-isomers of V. Heating IV with 90% H_2SO_4 20 min. at 100-10° gave 90% $HO_3SC_6H_4(OMe)CH_2CH_2CO_2H$, isolated as the *Ba salt*. Heating $PhCH_2CH:CCl_2$ with 92% H_2SO_4 20-30 min. at 55-60°, followed by standing overnight in the cold, gave 95% *sulfophenylpropionic acid*, purified through the Ba salt, which was then passed through an ion-exchange resin, yielding the free acid $HO_3SC_6H_4CH_2CH_2CO_2H$ *monohydrate*, hygroscopic crystals (from H_2O). G. M. Kosolapoff

2/2

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Freydlina, R. Kh.

USSR/Chemistry - Synthesis

Card 1/2 Pub. 40 - 6/27

Authors : Nesmeyanov, A. N.; Zakharkin, L. I.; and Freydlina, R. Kh.

Title : Derivation and dissociation constants of carboxylic acids

Periodical : Izv. AN SSSR. Otd. khim. nauk 1, 40-47, Jan-Feb 1955

Abstract : Data are presented regarding the synthesis of $CCl_3(CH_2)_nCOOH$ and $CCl_2CH(CH_2)_nCOOH$ carboxylic acids and their potentiometrically measured dissociation constants. It was found that the CCl_3- and CCl_2 = CH-groups have a definite effect on the dissociation constants of the acids which drop monotonously as these groups depart slowly from the carboxyl.

Institution : Acad. of Sc., USSR, Inst. of Elementary-Organic Compounds.

Submitted : March 31, 1954

APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3"

"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

Card 2/2 Pub. 40 - 6/27

Periodical : Izv. AN SSSR, Otd. khim. nauk 1, 40-47, Jan-Feb 1955

Abstract : It is shown that γ, γ dichlorovinylacetic acid has a higher dissociation constant that γ, γ, γ trichlorobutyric acid. The effects of the CCl_3 and CF_3 groups in higher ω, ω, ω,-trihalide carboxylic acids on the dissociation constants are described. Eleven references: 5 USA, 4 USSR and 2 German (1901-1954).

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R. KH.

USSR/ Chemistry - Organic chemistry

Card 1/1 Pub. 40 - 6/26

Authors : Freydlina, R. Kh.; Kost, V. N.; and Nesmeyanov, A. N.

Title : Chlorination and dehydrochlorination of polychloro derivatives containing reactive groups

Periodical : Izv. AN SSSR. Otd. khim. nauk 2, 233 - 239, Mar-Apr 1955

Abstract : Investigations were made to determine the reaction of chlorine with compounds containing the dichlorovinyl group and the reaction of dehydrochlorination of the dichloro-derivatives obtained. The results obtained during the chlorination of polychloro derivatives in hydrochloric acid media and dehydrochlorination under the effect of an alcohol solution of caustic potash are described. Eleven references: 5 USA, 1 English, 1 Belgian and 4 USSR and Russian (1884-1954).

Institution : Acad. of Sc., USSR, Inst. of Organoelemental Compounds

Submitted : June 3, 1954

APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3"

"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R. KH.

USSR/ Chemistry - Organic chemistry

Card 1/2 Pub. 22 - 22/50

Authors : Freydlina, R. Kh., and Vasilyeva, E. I.

Title : Effect of nitric acid on saturated polychloro hydrocarbons conteining the trichloromethyl group

Periodical : Dok. AN SSSR 100/1, 85-87, Jan 1, 1955

Abstract : It was established experimentally that nitric acid of specific weight 1.51 - 1.52 reacts with saturated polychloro hydrocarbons containing the trichloromethyl group already at room temperature resulting in the formation of hydrogen chloride. The results obtained from the reaction of nitric acid with alpha, alpha, alpha, omega-tetrachloroalkanes containing 5,7,9 and 11 carbon atoms in the molecule are listed It was found that compounds containing Cl in alpha-position relative to the

Institution: Acad. of Sc., USSR., Institute of Elementary Organic Compounds

Presented by: Academician A. N. Nesmeyanov, June 16, 1954

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

Periodical : Dok. AN SSSR 100/1, 85-57, Jan 1, 1955

Card 2/2 : Pub. 22 - 22/50

Abstract : trichloromethyl group experience a hydrolysis during their heating with fuming HNO_3. The trichloromethyl group converts into the carboxyl group when the reaction mixture is heated at 80-90° for a period of several hours. Nine references: 6 USA, 1 French, 1 German and 1 USSR (1893-(1954).

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FREYDLINA, R. Kh.

3

Chlorination of compounds containing the $CCl_2:CH$ group in sulfuric acid medium. *New method of synthesis of α-chloro carboxylic acids.* A. N. Nesmeyanov, V. N. Kost, and R. Kh. Freidlina (Inst. Heterogr. Compds. Acad. Sci., U.S.S.R., Moscow). *Doklady Akad. Nauk S.S.S.R.* 103, 1029-32(1955).—Chlorination of 1,1-dichloro-1-alkenes in H_2SO_4 at 0-20° results in formation of products, which after hydrolysis yield α-Cl acids (b.p./mm., n_D^{20}, and d_{20} given for the compds. reported below). Presumably the intermediate formed is $RCHClCCl_2OSO_3H$. Passage of Cl into 90 g. $Cl(CH_2)_3CH:CCl_2$ in 130 g. 93% H_2SO_4 at 5-20° until HCl evolution ceased, diln. with H_2O, extn. with $CHCl_3$, extn. of the latter with 10% NaOH, and acidification, gave 76% $Cl(CH_2)_3CHClCO_2H$, 106-7°/1, 1.4[illegible]5, 1.3121; *acid chloride*, 80°/5, 1.4840, 1.3513; *anilide*, m. 58-9°. Among other products found were: starting material and 10 g. $Cl(CH_2)_3CHClCCl_3$, 86-7°/2, 1.5100, 1.48[illegible]. Chlorination of 14 g. $PrCH:CCl_2$ in 26 g. H_2SO_4 at 5-1[illegible] similarly gave after aq. treatment 71% *$PrCHClCO_2H$*, 9[illegible]-4°/5, 1.4442, 1.1445; *acid chloride*, 61-2°/28, 1.4465, 1.170[illegible]; *anilide*, m. 63-4°. $Cl(CH_2)_3CH:CCl_2$ similarly gave 70% $Cl(CH_2)_3CHClCO_2H$, 128-30°/1, 1.4804, 1.2441, m. 22-4[illegible]; *acid chloride*, 104°/2, 1.4817, 1.2557; *anilide*, m. 42-3°. $C_6H_{11}CH:CCl_2$ gave 51% $C_6H_{11}CHClCO_2H$, 92-3°/1, 1.448[illegible], 1.0830; *chloride*, 76-7°/13, 1.4498, 1.1006. $Cl_2C:CH(CH_2)_3CO_2H$ gave 77% *2-chloroadipic acid*, m. 101-5°. $Cl_2C:CH(CH_2)_4CO_2H$ gave 73% *2-chloropimelic acid*, m. 88-9°. $Cl_2C:H(CH_2)_6CO_2H$ gave 69% *2-chlorosuberic acid*, m. 98-9°. Cf. *C.A.* 50, 4767g. G. M. Kosolapoff

9

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FREYDLINA, R. Kh.

Matls

Separation of aminocarboxylic acid from ammonium chloride with cation exchange resins. E. I. Basil'eva and R. Kh. Freidlina (*Izv. Akad. Nauk SSSR* [illegible] 150). — Effective separation of the acid from chlorides [illegible] be achieved, using suitable resins. The [illegible] 35–40 × 2 cm with 35–40 [illegible] 70% 6 aminocarboxylic [illegible] elution speed of 1–2 ml/min/cm² [illegible] at elution speed of 1·5 ml/min/cm² than at increased rates. No difference in effect was observed when NH_4 [illegible] 5 to 10%. Eluted amino acid 95–99% [illegible] separation of α and ω aminocarboxylic [illegible] in the same way.

2

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60. Research on Telomerization Described

"Investigation of the Reaction of Telomerization of Ethylene With Carbon Tetrachloride and the Chemical Transformations of alpha, alpha, alpha, omega-Tetrachloroalkanes" by A. N. Nesmeyanov, R. Kh. Freydlina, L. I. Zakharkin, Ye. I. Vasil'yeva, R. G. Petrova, Sh. A. Karapetyan, G. B. Ovakimyan, A. A. Beer, and M. A. Besprozvannyy, Khimicheskaya Pererabotka Neftyanykh Uglevodorodov (Chemical Conversion of Petroleum Hydrocarbons), Academy of Sciences USSR, Moscow, 1956, pp 303-323

APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3"

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It is pointed out that higher alpha, omega-bifunctional compounds such as glycols, diamines, dicarboxylic acids, aminocarboxylic acids, and hydroxycarboxylic acids, are of great importance as starting materials for the manufacture of a number of synthetic industrial products such as plastics, synthetic fibers, plasticizers, lubricating oils, and that for this reason the development of industrial methods for the synthesis of starting materials of this type from natural gas and industrial gases is an important undertaking. In view of the fact that telomerization reactions offer new possibilities for the synthesis of such materials, reactions of this type are now being investigated.

The results of experimental work on the following subjects are described: telomerization of ethylene with carbon tetrachloride in an autoclave (batch conversion); synthesis of higher alpha, alpha, alpha, omega-tetrachloralkanes; initiation of the telomerization reaction with short-wave radiation (X rays and gamma-rays emitted by Co^{60}), and chemical conversions of alpha, alpha, alpha, omega-tetrachloroalkanes including synthesis of omega-aminocarboxylic acids (omega-aminoenanthic

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acid, omega-aminopelargonic acid, and 11-aminoundecanoic acid), synthesis of beta-alanine from tetrachloropropane, synthesis of thiodicarboxylic acids of the constitution $S[(CH_2 - CH_2)_n COOH]_2$, and synthesis of normal dicarboxylic acids (including higher dicarboxylic acids such as 1,10-decanedicarboxylic acid and 1,14-tetradecanedicarboxylic acid).

With reference to the synthesis of thiodicarboxylic acids, the statement is made that these acids and their sulfones have been investigated thoroughly during recent years from the standpoint of their application in polycondensation processes. In connection with the telomerization of ethylene with carbon tetrachloride, a method of conducting this reaction continuously with recirculation of the unused ethylene is described; the batch method of reacting the mixture in an autoclave is stated to be dangerous because of the possibility of explosions. The following conclusions are given at the end of the paper:

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"The chemical transformations of alpha, alpha, alpha, omega-tetrachloroalkanes that are described in the paper are merely examples illustrating the profuse possibilities which are opened up by this type of synthesis. A review of other reactions of tetrachloroalkanes and trichloroalkanes has been published by A. N. Nesmeyanov, R. Kh. Freydlina, and L. I. Zakharkin in Uspekhi Khimii, Vol 25, No 6, June 1956, page 655. One must emphasize that a number of substances described in the present paper are of exceptional interest from the practical standpoint. Specifically, omega-aminocarboxylic acids are excellent starting materials for the synthesis of polyamide fibers.

"The fiber enant, which is derived from omega-aminoenanthic acid, is not inferior in its characteristics to other polyamide fibers such as capron and nylon. As far as a number of properties is concerned, e.g., thermal stability, stability to light, and elasticity, enant surpasses other polyamide fibers. The telomerization of ethylene with carbon tetrachloride, the conversion of 1,1,1,7-tetrachloroheptane into omega-aminoenanthic acid, and the conversion of 1,1,1,5-tetrachloropentane into delta, delta prime-thiodivaleric acid have been carried out at experimental [semiplant] installations." (U)

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREYDLINA, R. Kh.

Chlorination of unsaturated polychloro derivatives in acid medium. R. Kh. Freidlina, V. N. Kost, and A. N. Nesmeyanov (Inst. Heteroorg. Compds., Moscow). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1956, 1202-7; cf. *C.A.* 50, 4767g.—Passage of Cl until satn. into 26 g. 1,1,5-trichloro-1-pentene in 15 ml. Et_2O and 15 ml. concd. HCl satd. with HCl gave 81% *1,1,1,2,5-pentachloropentane*, $b_{1.6}$ 92-3°, n_D^{20} 1.5104, d_{20} 1.4815. Similar chlorination of 6,6-dichloro-5-hexenyl cyanide, followed by refluxing of the product 6 hrs. with 1:2 AcOH gave 80% *5,6,6,6-tetrachlorocaproic acid*, m. 47-8°. Similar chlorination of 8,8-dichloro-7-octenoic acid gave 99% *7,8,8,8-tetrachlorocaprylic acid*, b_1 158-60°, n_D^{20} 1.5018, which with concd. HNO_3 hydrolyzed to $HO_2C(CH_2)_4CHClCO_2H$, m. 98-9°. Passage of Cl into 18 g. 1,1,5-trichloro-1-pentene in 20 g. 90% H_3PO_4 at 0-10° gave only polychlorohydrocarbons, but a similar reaction run at 30° in 70% $HClO_3$, followed by 2 hrs. on a steam bath gave 36% $CH_2Cl(CH_2)_2CHClCO_2H$, b_1 105-8°, n_D^{20} 1.4859, and neutral products which were not investigated owing to their explosive nature. Condensation of 3,3,3-trichloropropene with 1,1-dichloroethylene (cf. Nesmeyanov, *et al.*, *C.A.* 49, 60093g) gave a mixt. of products from which was fractionated *1,1,5,5,5-pentachloropentene*, $b_{1.6}$ 71°, n_D^{20} 1.5108, d_{20} 1.4771, and *1,1,5,5-tetrachloro-1,4-pentadiene*, $b_{1.6}$ 67°, n_D^{20} 1.51[illegible] [illegible] The former (62 g.) in 75 g. 93% H_2SO_4 was [illegible] 20-6° yielding after diln. with H_2O 52% *2,5,5,5-tetrachloropentanoic acid*, m. 82-3°. Passage of Cl at 0-10° into 21 g.

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Fridlion, R.Kh.; V.N.Kost; Nesmeyanov, A.N.

2,3,3-trichloropropene in 20 g. concd. H_2SO_4 gave 14 g. *1,1,1,2,3-pentachloropropane*, b_4 54-6°, n_D^{20} 1.5100, and 11 g. *di-2,3,3,3-tetrachloropropyl sulfate*, b_4 189 [illegible] the latter hydrolyzing in acid medium [illegible] *propanol* (I), m. 39-40°. The same [illegible] AcOH gave largely 1,1,1,2,3-pentachloropropane [illegible] smaller yield of *1,1,1,2-tetrachloro-3-acetoxypropane*, and a n_D^{20} 1.4368, d_4^{20} 1.4905; the latter with HCl-MeOH gave I. Refluxing 58 g. 2,3,3,3-trichloropropene with 40 g. NaOAc and 75 ml. AcOH 12 hrs. gave 43 g. *1,1-dichloro-3-acetoxy-1-propene*, b_4 71-2°, n_D^{20} 1.4850, d_4^{20} 1.2840, which with MeOH-HCl gave *3,3-dichloro-2-propen-1-ol*, b_4 66-7°, n_D^{20} 1.4945, d_4^{20} 1.3763, which (25 g.) in 25 ml. HCl and 25 ml. Et_2O was satd. with HCl and treated with Cl at −5 to 0° yielding 65% *2,2,3,3-tetrachloro-3-propanol*, b_4 76°, m. 39-40°, and a higher-boiling residue; if the chlorination is run in $CHCl_3$, much HCl is evolved and only 21% addn. product is isolated. Chlorination of *1,1-dichloro-3-methoxy-1-propene* (loc. cit.) gave 62% *pentachloro deriv.*, $b_{4.5}$ 81-3°, n_D^{20} 1.5070, d_4^{20} 1.5713, which refluxed with MeOH-HCl gave 90% *2,2,3,3-tetrachloro-1-propanol*, m. 38-9°, identical with the above specimen; hence the pentachloro deriv. is $CCl_2CHClCH_2OCH_2Cl$, also formed in 50% yield from paraformaldehyde with HCl in $CHCl_3$ and 2,3,3,3-tetrachloro-1-propanol. Thus, conjugated addn. to terminally halogenated olefins occurs only in the medium of strong concd. acids.

G. M. Kosolapoff

2/2

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

NESMEYANOV, A.N.; NOGINA, O.V.; FREYDLINA, R.Kh.

Preparation of hexaalkyloxydititanoxanes $(RO)_3Ti -- O -- Ti(OR)_3$. Izv.AN SSSR.Otd.khim.nauk no.3:373-375 Mr '56. (MLRA 9:8)

1. Institut elementoorganicheskikh soyedineniy Akademii nauk SSSR.
(Titanium organic compounds)

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

FREIDLINA, R.KH.

Chem

Tetra- and trichloroalkanes and related compounds. A. N. Nesmeyanov, R. Kh. Freidlina, and [illegible] (U.S.S.R. Acad. Sci., Moscow) [illegible] 330-70(1956).—Review, 86 references. R. H.

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FREYDLINA, R. KH.

USSR/Organic Chemistry - Synthetic Organic Chemistry, E-2

Abst Journal: Referat Zhur - Khimiya, No 1, 1957, 800

Author: Nesmeyanov, A. N., Freydlina, R. Kh., and Zakharkin, L. I.

Institution: None

Title: The Investigation of the Reactions of Polychlorohydrocarbons and Related Compounds

Original Periodical: Uspekhi khimii, 1956, Vol 25, No 6, 665-704

Abstract: A detailed survey with a bibliography of 93 titles.

Card 1/1

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"APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000513710001-3

USSR/Organic Chemistry - Theoretical and General Questions on Crganic Chemistry E-1

Abs Jour : Referat Zhur - Khimiya, No 2, 1957, 4228

which is attributed to spatial hindrances. In an alcoholic medium $(C_2H_5)_2NH$ reacts with V and VI to form mixtures of alkoxy- and amino- derivatives, while with VII and VIII only alkoxy-derivatives are formed. With $(C_2H_5)_2NH$ alone, without alcohol, V and VI form amino-derivatives, while VII does not react even at 110°. On action of NH_3 and piperidine (XL), in an alcoholic medium, VII forms a small amount of amino-derivatives. The same course of the reaction has been ascertained on interaction of V, VI and VII with Na_2S in alcoholic medium: V and VI form mixtures of O- and S-derivatives, while VII yields only O-derivatives. The authors draw the conclusion that the reactivity of the investigated compounds depends to a large extent upon the nature of the substituents at the center of action of the NP. With decrease of the electrophilic nature (EP) of the compound under

Card 3/14

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Abs Jour : Referat Zhur - Khimiya, No 2, 1957, 4228

on interaction of III with alcoholic KOH there is formed, in addition to VII, 1,1-dichloro-3-methyl-butadiene-1,3 (XV). Action of excess alkali in III or on 1,1,1,3-tetrachloro-3-methylbutane (XVI), in CH_3OH or C_2H_5OH, gives 1,1-dichloro-3-methoxy- (XVII) or 1,1-dichloro-3-ethoxy-3-methylbutene-1 (XVIII). On heating XVIII is converted to XV. VII also forms XV on boiling and in CH_3COCH at 100°. On action of HCl (gaseous) XV and XVII form X, which is readily reconverted to XV (alcoholic alkali). It is shown that on interaction of VII with alcoholic alkali, $(C_2H_5)_2NH$ or XI, there is formed an admixture of XV. The assumption is made that XV can be formed by isomerization of VII to X and subsequent splitting off of HCl or as a result of direct action of

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Abs Jour : Referat Zhur - Khimiya, No 2, 1957, 4228

64%, BP 91-92°/1 mm, $n^{20}D$ 1.5710, d_4^{20} 1.3217. On dehydrobromination with alkali in C_2H_5OH VI reacts with alcohol. 153 g III in 50 ml alcohol treated with 45 g KOH in 180 g alcohol (0°) to get XV, yield 3.5 g, BP 30-31°/8 mm, $n^{20}D$ 1.5027, and VII, yield 60.5%, BP 45-46°/8 mm, $n^{20}D$ 1.4822, d_4^{20} 1.2497. From 63 g XVI and 47 g KOH in 150 ml CH_3OH (15-20°, 1 day) are obtained XV, yield 5.1 g, and XVII, yield 71%, BP 51-52°/10 mm, $n^{20}D$ 1.4628, d_4^{20} 1.1418. In the same manner from 50 g XVI, 35 g KOH and 100 ml C_2H_5OH are obtained 12 g XV and XVIII, yield 14 g, BP 72-73°/24 mm, $n^{20}D$ 1.4616, d_4^{20} 1.1101. 17 g XVII and C_2H_5ONa (from 6 g Na) in 80 ml absolute alcohol boiled 10 hours, added 8 g KOH in 10 ml water and boiled 6 hours, to get $(CH_3)_2$-C:CHOOH, yield 4.2 g, MP 69-70° (from water). Into 39.5 g XVII HCl (gas) is passed for 2.5 hours, stratified liquid is poured into HCl, bottom layer is separated,

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Abs Jour : Referat Zhur - Khimiya, No 2, 1957, 4228

and therefrom is obtained X, yield 30.1 g, BP 58-58.5°/15 mm, $n^{20}D$ 1.4847, d_4^{20} 1.2527. X is also obtained by passing HCl (gas) (-5 to -7°, 3 hours) into 14 g XV in presence of 1 g $ZnCl_2$, yield 52%, BP 64°/25 mm. From 26 g 4,4-dimethylbutene-1, 60 g $CBrCl_3$ and 1 g of dinitrile of azo-bis-isobutyric acid (autoclave, 100-105°, 6 hours) is obtained IV, yield 38 g, BP 90°/5 mm, $n^{20}D$ 1.5030, d_4^{20} 1.4792. From 0.11 mole IV and 0.33 mole $(C_2H_5)_2NH$ (~ 100°, 18 hours) is synthesized VIII, yield 70%, BP 64-65°/10 mm, $n^{20}D$ 1.4725, d_4^{20} 1.1403. Ozonation of VIII yields the chloral. From 35.6 g V and CH_3ONa (from 4.98 g Na in 50 ml CH_3OH) is obtained (~ 100°, 2.5 hours) 1,1-dichloro-3-methoxybutene-1 (XX), yield 50%, BP 64°/57 mm, $n^{20}D$ 1.4580, d_4^{20} 1.1722. 15.9 g V and 18.3 g $(C_2H_5)_2NH$ (boiled 8 hours) form 1,1-dichloro-3-diethylamino-butene-1 (XXI), yield 50%,

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USSR/Organic Chemistry - Theoretical and General Questions on Organic Chemistry E-1

Abs Jour : Referat Zhur - Khimiya, No 2, 1957, 4228

-3-methyl-2-carbethoxypentene-4-ic acid (XXIII), yield 70%, BP 107°/1 mm, $n^{20}D$ 1.4605, d_4^{20} 1.1829. 20.7 g XXIII are hydrolyzed (by boiling with HCl and CH_3COCH) and decarboxylated to 5,5-dichloro-3-methylpentene-4-ic acid, yield 85.7%, BP 102°/1 mm, $n^{20}D$ 1.4800, d_4^{20} 1.2739. By interaction of 10 g V, 1.2 g $AlCl_3$ and 25 ml C_6H_6 (stirred 30 minutes) is obtained XII, yield 70%, BP 73-74°/ 1.5 mm, $n^{20}D$ 1.5423, d_4^{20} 1.1702. On action of Cl_2 XII forms 1,1,1,2-tetrachloro-3-phenylbutane, BP 107-108°/1.5 mm, $n^{20}D$ 1.5568, d_4^{20} 1.3634. From 36 g IX and 36.5 g $(C_2H_5)_2NH$ (~ 100°, 16 hours) are obtained XXI, yield 13.8 g, BP 84-87°/21 mm, and 20.6 g IX. Interaction of 15 g VI and 5.5 g KOH in 25 ml absolute C_2H_5OH (15-20°, 16 hours) gives 1,1-dichloro-3-ethoxy-3-phenylpropene-1 (XXIV), yield 87%, BP 90-91°/1 mm, $n^{20}D$ 1.5308, d_4^{20} 1.1822. From 11 g VI and 12 g

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Synthesis of higher γ,δ,ω-tetrachloroalkanes and 1,1,1-trichloroalkanes. A. N. Nesmeyanov, Sh. A. Karapetyan, and K. Kh. Freidlina (Inst. Hetero-org. Compds. Acad. Sci. U.S.S.R., Moscow). *Doklady Akad. Nauk S.S.S.R.* 109, 791-3(1956).—Reaction of C_2H_4 with CCl_4 in an autoclave with 0.8-1 g isobutyroazobisnitrile catalyst per l. was studied at 92° with 4-5-hr. runs. The reaction gave up to 64% C_4 tetrachloroalkanes, up to 28% C_7 homologs, and up to 70% C_7 or higher homologs. The latter are favored by higher proportions of the olefin component. The results were similar with $CHCl_3$-ethylene mixts., yielding up to 54% C_{12} and higher trichloroalkanes. The effect on yields of various fractions exerted by the change of compn. of the charge is shown graphically and in each case the yield of any fraction passes through a max. The following were isolated in pure state: *1,1,1-trichlorotridecane*, $b_{0.1}$ 103-8°, d_{20} 1.0339, n_D^{20} 1.4640; *1,1,1-trichloropentadecane*, $b_{0.1}$ 123-5°, d_{20} 1.0142, n_D^{20} 1.4658, and *1,1,1-trichloroheptadecane*, $b_{0.1}$ 138-43°, d_{20} 0.9902, n_D^{20} 1.4663.

G. M. Kosolapoff

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FREYDLINA, R. Kh.

Condensation of γ,γ,γ-trichlorocrotonic, γ,γ-dichlorocrotonic, and γ-bromocrotonic acids with benzene in the presence of aluminum chloride. A. N. Nesmeyanov, L. I. Zakharkin, and R. Kh. Freidlina (Inst. Hetero-org. Compds., Moscow). *Doklady Akad. Nauk S.S.S.R.* 111, 114-16(1956).—Adding 18 g. $AlCl_3$ to 20 g. $CCl_3CH:CHCO_2H$ in 80 ml. C_6H_6, stirring 2 hrs. at 35-45°, and treating with aq. HCl gave 18.1 g. *$CCl_2:CHCHPhCO_2H$* (I), $b_{1.5}$ 145-6°, m. 87-8°; this hydrogenated over Pd-$BaSO_4$ in aq. NaOH gave the Cl-free acid, yielding with $SOCl_2$ an acyl chloride which with NH_3 gave *$PhEtCHCONH_2$*, m. 84-5°. Heating I with Et_3N 3 hrs. at 50° and treating with aq. HCl gave a fair yield of PhCH:CHCHO. Reaction of 15.5 g. $CHCl_2CH:CHCO_2H$, 60 ml. C_6H_6, and 15 g. $AlCl_3$ 1 hr. at 40° similarly gave 18.8 g. *$PhCHCH:CHCO_2H$* (II), b_2 185-6°, m. 135-6°; heated with aq. MeOH-KOH 3 hrs., this gave *$Ph_2C:CHCH_2CO_2H$*, m. 115-16°, while oxidation with $KMnO_4$ yielded Ph_2CHCO_2H, thus confirming the structure of II. $CH_2ClCH:CHCO_2H$ (16 g.), 60 ml. C_6H_6, and 15 g. $AlCl_3$ similarly gave 10.2 g. *$PhCH_2CH:CHCO_2H$*, b_2 137-8°, m. 64-5°. G. M. Kosolapoff

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FREYDLINA, R. Kh.

Carboxylic acids of the fatty and fatty aromatic series. R. Kh. Freidlina and R. L. Vasil'eva. U.S.S.R. 105,125, Mar. 25, 1957. The carboxylic acids are obtained by hydrolyzing the corresponding polychlorohydrocarbons contg. a Cl_3C or dichlorovinyl group. As hydrolyzing agent is used 70% $HClO_4$. M. Hosch

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ω-Aminoacanthic acid. R. Kh. Freidlina and L. I. Zakharkin. U.S.S.R. 105,269, May 25, 1957. Tetrachloroheptane is aminated with NH_4 in MeOH with heating and the resulting aminotrichloroheptane is sapond. by heating it with concd. H_2SO_4. Another method is to convert tetrachloroheptane into chloroheptanoic acid by usual means and then aminate the acid by heating with 25% NH_4OH under pressure. M. Hosch

11
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α,α,α,ω-Tetrachloroalkanes. A. N. Nesmeyanov, R. Kh. Freidlina, L. I. Zakharkin, A. Kh. Khomenko, A. H. Belyavskii, G. B. Ovakimyan, A. A. Beer, and M. A. Besprosvanyi. U.S.S.R. 105,655, July 25, 1957. The title compd. is obtained by the interaction of ethylene and CCl_4. To reduce explosion hazards, the reaction is carried out in a flow type reactor in the presence of Bz_2O_2 or azodinitrile of diazo butyric acid or some other initiator which decomp. at 50-100°. The reaction can be also initiated by ultraviolet irradiation at 30-40°. M. Hosch

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FREYDLINA, R. S., NESMEYANOV, A.N., SAKHARKIN, L. U., VASILYEVA, [illegible] Y., [illegible], [illegible] G., and KARAPETYAN, S. A.

"Polymerization of ethylene with telomers and a new synthesis of amino acids," a paper presented at the 9th Congress on the Chemistry and Physics of High Polymers, 28 Jan-2 Feb 57, Moscow Organic Chemistry Research Inst.

B-3,084,395

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FREYDLINA, R.Kh., FREYDLINA, R., NESMEYANOV, A.N., KOST, V.N., ZAHHARKIN, L.I.

AS USSR

"Homolytic Isomerisation in Polyhalogenalkenes," paper submitted at 16th International Congress of Pure and Applied Chemistry, Paris, 18-24 July 1957

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FREYDLINA, R. Kh.

7

Preparation of α-proline and α-ornithine from 1,1,1,5-tetrachloropentane. A. N. Nesmeyanov, R. Kh. Freidlina, and R. G. Petrova (Inst. Heteroorg. Compds., Moscow). *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1957, 451-8.—Keeping 15 g. $ClCH_2CH_2CH_2CHClCO_2H$ 72 hrs. at 20-25° in 350 ml. 25% NH_4OH, removing NH_3 by heating, passing the residual soln. over a cationic resin, washing the resin with H_2O, and eluting with 5% NH_4OH gave on evapn. 25.5% α-proline, m. 202-3° (from EtOH-dioxane); the filtrate on evapn. and treatment with $CuCO_3$ gave addnl. 28.4% proline Cu salt; the total yield was 53.9%. Similar reaction of $ClCH_2CH_2CH_2CHBrCO_2H$ in 48 hrs. gave 10% proline and 41.3% its Cu salt, for a total yield of 51.5%. Heating 30 g. $ClCH_2CH_2CH_2CHClCO_2Et$ and 16.5 g. NH_3 in 90 ml. EtOH in an autoclave 3 hrs. at 130° and heating the crude product several hrs. with concd. HCl gave 31% proline. Heating 12.3 g. $CCl_2CHClCH_2CH_2CH_2Cl$, 24 g. NH_3, and 150 ml. EtOH 3 hrs. at 100° gave 41.6% insol. ppt. of $CCl_2:CClCH_2CH_2CH_2NH_2.HCl$, which with NaOH gave the *free amine*, b_5 70-1°, n_D^{20} 1.5120, d_{20} 1.3180. Heating 12 g. $CCl_2CHClCH_2CH_2CH_2Cl$, 23 g. o-$C_6H_4(CO)_2NK$, and 25 ml. abs. EtOH 6 hrs. at 185-90° gave 61.3% *1,1,2-trichloro-5-phthalimido-1-pentene*, m. 87-8° (from EtOH). Passage of Cl into 12.5 g. $CCl_2.CHCH_2$-

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Nesmeyanov, A.N.; Freidlina, R.Kh.; Petrova, R.G.

$CH_2CH_2NH_2$ at 0° followed by diln. with 40 ml. H_2O and neutralization with NH_4OH gave 0.75 g. *$CCl_3CHClCH_2CH_2CH_2NH_2.HCl$*, decomp. 105-7°, while the aq. filtrate gave 0.5 g. *$H_2NCH_2CH_2CH_2CHClCO_2H$*, m. 138°. The filtrates from both substances were passed over a cationic resin and eluted with 5% NH_4OH to yield after treatment with $CuCO_3$ 41.5% proline Cu salt. Heating 1.3 g. $CCl_3CHClCH_2CH_2CH_2NH_2$ and 5 g. *o-$C_6H_4(CO)_2O$* at the m.p. several hrs. gave 75% *1,1-dichloro-5-phthalimido-1-pentene*, m. 103-7°, which formed in 75% yield on heating 70 g. *o-$C_6H_4(CO)_2NK$*, 65 g. $CCl_3CHClCH_2CH_2CH_2Cl$, and 20 ml. abs. EtOH in an autoclave 12 hrs. at 185-95°. The product (9 g.) in 40 ml. concd. H_2SO_4 was chlorinated at 0° 3 hrs. then quenched in H_2O yielding 92% *2-chloro-5-phthalimidopentanoic acid*, m. 118-19° (from C_6H_6). This (30 g.) in 125 ml. 25% NH_4OH was heated in an autoclave with 135 g. $(NH_4)_2CO_3$ and 45 ml. H_2O 15 hrs. at 100° yielding 33% DL-ornithine HCl salt after heating the crude product with concd. HCl 12 hrs., sepg. the phthalic acid, evapg., pptg. the NH_4Cl with EtOH, and adding NH_4OH to pH 6-7. The filtrate from the ornithine-HCl was concd. and treated with isatin yielding 31.2% DL-proline-isatin condensation product.

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FREYDLINA, R. Kh.

7

Reaction of 1,1,1-trichloropropene and 1,1,1,3-tetrachloropropane with aromatic amines. R. Kh. Freidlina and N. A. Semenov. (Inst. Heteroorg. Compds., Moscow). Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk 1957, 466-70; cf. C.A. 50, 11278h.—Refluxing 75 g. $CCl_3CH:CH_2$ and 325 g. $PhNH_2$ 1 hr., treating with satd. Na_2CO_3, and extg. with Et_2O gave 77% *$PhNHCH_2CH:CCl_2$* (I), b_4 122-3°, n_D^{20} 1.5873, d_{20} 1.2522 (*HCl salt*, m. 144-5°), and 5% *$PhN(CH_2CH:CCl_2)_2$*, $b_{0.4}$ 156-7°, 1.5845, 1.3516 (*HCl salt*, m. 139-40°). The latter formed in 65% yield on heating the former with excess $CCl_3:CHCH_2Cl$. Heating I with excess concd. H_2SO_4 1 hr. at 120-45°, adding 60 ml. EtOH, heating 20 min. at 80-90°, pouring the mixt. into concd. Na_2CO_3, and extg. with Et_2O gave 80% *$PhNHCH_2CH_2CO_2Et$*, b_3 146-7°, 1.5313, 1.0679; *HCl salt*, m. 68-9°. Refluxing 15 g. I with 30 g. Ac_2O 1 hr. gave *$PhNAcCH_2CH:CCl_2$*, b_4 138-9°, 1.5550, 1.2399, which heated with 94% H_2SO_4 1 hr. at 100° and 0.5 hr. at 140°, quenched in ice, and extd. with $CHCl_3$ gave 90% *$PhAcNCH_2CH_2CO_2H$*, m. 110-11° (from H_2O). Refluxing $CCl_3CH:CH_2$ and 137 g. $PhNHMe$ 1 hr., adding satd. Na_2CO_3, and extg. with $CHCl_3$ gave 76% *$PhNMeCH_2CH:CCl_2$* (II), b_4 120°, b_6 135°, 1.5800, 1.2142; *HCl salt*, m. 126°. Refluxing 68 g. $CCl_3CH:CH_2$ and 240 g. $PhNMe_2$ 4 hrs. gave 20 g. MeCl and 85% II. Refluxing 112 g. $CCl_3CH_2CH_2Cl$ and 281 g. $PhNH_2$ 2 hrs. gave 20 g. yellowish substance (III), b_4 127-8°, 1.5765, 1.3182, which decompd. violently on attempted distn. with Zn dust; treated with EtONa it gave *$PhNHCH_2CH:CCl_2$*. While isolation of III was difficult, its *HCl salt*, *$PhNHCH_2CH_2CCl_3.HCl$*, m. 170-1°, was readily obtained by passage of dry HCl into its soln.; this heated with Ac_2O gave *Ac deriv.*, b_4 154°, 1.5460, 1.3054, m. 47° (on

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FREIDLINA, R. KH.; SEMENOV, N. A.

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standing). The latter treated with an equimolar amt. of EtONa in EtOH gave $PhAcNCH_2CH:CCl_2$, identical with that described above; heated with concd. H_2SO_4 at 100–20° it gave *$PhAcNCH_2CH_2CO_2H$*, m. 110–10.5°. Refluxing 36 g. $CCl_2CH_2CH_2Cl$ and 44 g. PhNHMe 2 hrs. gave *$PhNMeCH_2CH_2CCl_3$*, b_6 125–35°, 1.5720, 1.2393; *HCl salt*, decomp. 178–9°; the latter with EtONa gave *$PhNMeCH_2CH:CCl_2$*, whose *HCl salt*, m. 128°, was identical with II. G.-M. Kosolapoff

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FREYDLINA, R. Kh.

4

Action of nucleophilic reagents on compounds of the type $[Cl(CH_2)_nCCl_3]_2$. 7%. Kh. Freydlina and T. A. Kost (Inst. Heterorg. Compds., Moscow). Izvest. Akad. Nauk S.S.S.R., Oldel. Khim. Nauk 1957, 644-6.—[The compds. of the type $[R(CH_2)_nCCl_3]$ in this abstr. are represented by A, followed in parentheses by R and n.] Refluxing A(Cl, 4) (I) with NaI in Me_2CO 10 hrs. gave 68% A(iodo, 4), m. 113-17° (from petr. ether); similarly A(Cl, 6) (II) gave 89.7% A(iodo, 6) (III), m. 94.5-5.5° (from EtOH), which with NaCN yielded A(cyano, 6), m. 93°. Heating 17.5 g. I with 14.7 g. KOAc and 1 g. KI in AcOH 10 hrs. gave 83% A(AcO, 4), m. 102-3°, which, refluxed with HCl-MeOH 5 hrs. yielded 86% A(HO, 4), m. 76-7°. I and NaCN in aq. EtOH 6 hrs. gave 90% A(cyano, 4), m. 63-4°; similarly II, NaCN, KI, and aq. $EtOCH_2CH_2OH$ heated 6 hrs. gave 93% A(cyano, 6), m. 75°, yielding with HCl-AcOH in 8 hrs. 82.7% A(HO_2C, 6), m. 177-8°. I (34.9 g.), 10.8 g. KOAc and 1 g. KI in 100 ml. AcOH in 18 hrs. at 120° gave 94% $Cl(CH_2)_n(CCl_2)_2(CH_2)_2OAc$, m. 46-6.5°. II heated with 4 moles NaI in Me_2CO 14 hrs. gave 78% $Cl(CH_2)_n(CCl_2)_2(CH_2)_2I$, m. 83-3.5°.

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FREYDLINA, R. KH.

AUTHORS: Freydlina, R. Kh.; Braynina, E. M.; Nesmeyanov, A. N. 62-1-6/21

TITLE: Interchange Reactions of Zirconium Tetraacetylacetonate (Obmennyye reaktsii tetraacetylatsetonata tsirkoniya)

PERIODICAL: Izvestiya Akademii Nauk SSSR, Otdeleniye Khimicheskikh Nauk, 1957, No. 1, pp. 43-47 (U.S.S.R.)

ABSTRACT: This report is devoted to the study of interchange reactiona of zirconium tetraacetylacetonate, which appears to be one of the easily accessible chelate compounds. The possibility of converting zirconium tetraacetylacetonate into other less-accessible chelate compounds of octo-coordination zirconium, was investigated. The conversion into hexacoordinated Zr compounds and into alkylorthozirconates was also studied. It was established that the reaction between zirconium tetraacetylacetonate and benzoyl acetone leads to an interchange between the radicals of one beta-diketone into radicals of another beta-diketone and the formation of zirconium tetrabenzoyl acetonate,

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Interchange Reactions of Zirconium Tetraacetylacetonate 62-1-6/21

ASSOCIATION: Academy of Sciences of the USSR, Institute of Elemento-Organic Compounds

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SUBMITTED: January 21, 1956

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FREYDLINA, R. Kh.

Homolytic isomerization of 1,1,1-trichloro-2-bromopropane. V. A. N. Nesmeyanov, R. Kh. Freidlina and V. N. Kost (Akad. Nauk S.S.S.R., Moscow). Tetrahedron 1, 241-7(1957).—The homolytic isomerization of CCl_3CBrCH_3 (I) to CCl_3CClCH_3 (II) [illegible] light was compared with the heterolytic antinomic anyne rearrangement of I to CCl_3CBrCH_3Cl (III) under the action of $SbCl_3$ or $AlCl_3$. $CCl_3CH(CH_3)$ (IV) [illegible] AcOH treated portionw[illegible] the [illegible] with [illegible] H_2O, aq. Na_2CO_3, and H_2O [illegible] 50% $CCl_3CHBrCH_3Br$ (V) [illegible] V (15 g.) in 35 cc. HNO_3 (d. 1.52) reduced [illegible] only traces of acidic material [illegible] $C_4H_7BrCl_3NO_2$, b. 79-80°, n^{20} 1.5[illegible] [illegible] 48 g. treated dropwise [illegible] [illegible] H_2O, the oil extr. and [illegible] distd. gave 25 g. I, b. 57-8°, d. 1.5[illegible], n^{20} 1.532[illegible] verted by heating 10 g. with 8.5 g. [illegible] lux on a steam bath to give [illegible] b. 50°, d. 1.4060, n^{20} 1.5070; [illegible] $CHCl_3$). Heating 16 g. I with 5[illegible] [illegible]

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NESMEYANOV, A.N., FREIDLINA, R.KH., KOST, V.N.

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$AlCl_3$ yielded 78% $PhCH_2CBr:CCl_2$, b_{12} 97–8°, d_{20} 1.5[illegible], n_D^{20} 1.5840. I (10 g.) treated with 0.2 g. $SbCl_3$ [illegible] and the soln. kept 2 hrs. at 20°, the [illegible] HCl and H_2O, dried, and distd. gave [illegible] g. III, [illegible] d_{20} 1.8954, n_D^{20} 1.5522, also produced by dehydrobromination of $CCl_2BrCHBrCH_2Cl$. On exposure to ultraviolet light, I isomerized completely to II, [illegible] 78–9°, d_{20} 1.8[illegible], n_D^{20} 1.5550, converted by treatment with $HNEt_2$ into $CCl_2:CClCH_2NEt_2$, $b_{2.5}$ 63°, d_{20} 1.1910, n_D^{20} 1.4885. II was also obtained by treating $CCl_2:CClCH_2Cl$ with NaBr in Me_2CO. Br (48 g.) in 50 cc. $CHCl_3$ added gradually to 48 g. $CCl_2:CHCH_2Cl$ in 50 cc. $CHCl_3$, the $CHCl_3$ evapd., and the residue distd. *in vacuo* gave 85 g. $BrCCl_2CHBrCH_2Cl$ (VII), d_{20} 2.1874, n_D^{20} 1.5678. VII (15 g.) in 30 cc. HNO_3 (d. 1.52) heated gently, the final reaction mixt. refluxed 30 min., dild. with H_2O, and extd. with $CHCl_3$, the ext. washed with aq. Na_2CO_3, the alk. washings acidified, the acid product extd. with $CHCl_3$, and the dried ext. distd. gave 7.2 g. $ClCH_2CHBrCO_2H$, b_{12} 125–6°, m. 43–4°, converted by alc. KOH to $H_2C:CBrCO_2H$, m. 68–9°. Dehydrobromination of 70 g. VII and fractional distn. of the product gave 22 g. $CCl_2:CBrCH_2Cl$ (VIII), b_{11} 81–2°, d_{20} 1.8955, n_D^{20} 1.5522, together with 13 g. $BrCCl:CBrCH_2Cl$ (VIIIa), b_{11} 97–8°, d_{20} 2.1957, n_D^{20} 1.5845. Treatment of VIII or VIIIa with $HNEt_2$ in MeOH gave VI. IV (36 g.) in 60 cc. $CHCl_3$ treated portionwise with 40 g. Br in 30 cc. $CHCl_3$ with stirring and exposure to electric light (150 w.), the $CHCl_3$

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NESMEYANOV, A.N. FREIJUNA R.Kh. KOST, Y.N.

evapd. and the residue [illegible]
of trichlorobiphenyl [illegible]
[illegible] The mixt. [illegible]
1.52) as above yielded [illegible]
[illegible]
[illegible]
[illegible]
[illegible]
BrCCl CH₃ (X [illegible]
[illegible]
[illegible]
(XII) [illegible]
cc CCl₄ treated dropwise with [illegible]
the solvent evapd., and the residue distd [illegible]
mixt. of trichlorobiphenyl [illegible]
1.5040) Oxidation [illegible]
[illegible]
[illegible]
[illegible]
[illegible]
[illegible]
[illegible]

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FREYDLINA, R.Kh.; CHUKOVSKAYA, Ye.TS.

Interreaction between mercuric acetate and xanthogenic acid esters. Izv.AN SSSR. Otd.khim.nauk no.2:187-193 F '57. (MLRA 10:4)

1. Institut elementoorganicheskikh soyedineniy Akademii nauk SSSR.
(Mercury acetates) (Xanthic acids)

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FREYDLINA, R.Kh, doktor khimicheskikh nauk; VASIL'YEVA, Ye.I., kandidat khimicheskikh nauk.

Reaction of homolytic telomerization. Khim. nauka i prom. 2 no.1:2-12 '57. (MIRA 10:4)
(Polymerization)

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FREYDLINA, R.Kh.

ω-Aminocarboxylic acids from carbon-tetrachloride-ethylene telomers. New polyamide fibers. [illegible]

A. A. St[illegible]
Vasil'ev[illegible]
Sh[illegible]
M[illegible]

[illegible]

with [illegible]
to give [illegible]
(CH_2CH_2[illegible]
II [illegible]
(CH_2[illegible]
the [illegible]
ably in various [illegible]
Conversion of I [illegible] CH_2[illegible]
prepd. [illegible]
stances [illegible]
stud[illegible]
consist[illegible]
decreasing [illegible]
negligi[illegible]
produ[illegible]
(65%) at [illegible] atm. and [illegible]
curring at [illegible] atm. [illegible]
atm.), the [illegible]
to 35%. At 90° 12[illegible]
of 1:4, CCl_4 utilizat[illegible]
reaches 47% at 120 min.; at 100°, 32% of the CCl_4 has reacted in 80 min. and further utilization is negligible. Using a 1:10 ratio of reactants [illegible] 0.8-1.0 g I/l CCl_4), CCl_4 utilization continues to rise, reaching 40% at 80°, and 65% at 90° after 120 min.; at 100°, little more CCl_4 reacts after [illegible] CH_2 CH_2-CCl_4 ratios [illegible] of higher pr[illegible] I, n > 3 are [illegible] to 61% ([illegible] conversion of [illegible] and 94% (38% conversion), resp.; removal of volatile products (including II, n = 7) left residues (55 and 75%, resp.) fractionated on the basis of CCl_4 and acetonesoly. Into materials of mean mol. wt. 420-840, all analys-

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Nesmeyanov, A.N.; Stepichkov, A.A.; [illegible]

ing well for $C_6H_{12-x}Cl_x$. Using 2[illegible] g CCl_4, 130 atm. CH_4; CH_4 pres[illegible] yields of

[illegible]

[illegible]uction [illegible] CCl_4 [illegible]

[illegible]

[illegible] group [illegible]

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Nesn [illegible] AN [illegible] A.A.; [illegible]

A, 93%, [illegible]

[illegible] (m. 62–3°); n = 14, A, 24%, —, —, — (m. 62–3°). Similarly obtained from IV are straight-chain fatty acids having an odd no. of C atoms: C_{11}, B, 70%, 93–4°/5, 1.4248, 0.9231; C_{13}, B, 69%, —, —, — (m. 33°); C_{15}, B, 67%, —, —, — (m. 5[illegible]°); C_{17}, B, [illegible], —, —, — (m. 61°). V is converted to III by several methods: D, heating V with 25% aq. NH_3 (50–60 ml./gr.) at 100° 1–2 hrs., venting excess NH_3, running the residual soln. through a resin column (SBS or SDW, washed free of Cl), eluting III with 5% aq. NH_3, stripping the eluate, extg. the residue (HNR, remains undissolved) with H_2O, decolorizing the [illegible] recrystg. the residual III [illegible] 5–7 [illegible] 24% [illegible] addn. of 4 [illegible] decomp. by heating [illegible] up as in D; G, heating [illegible] ml. H_2O 3 hrs. at 100°. III (n = 1) m. 196–7° (from H_2O, as are other examples of III), yield 90% (method E); III (n = 3), m. 195°, 67% (D), 85–90% (E), 81% (F), 80% (G); III (n = 4) m. 189–90°, 80% (D), 82% (E), 80% (F), 82% (G); III (n = 5) m. 185–6°, 89% by reaction with 40 equivs. 40% aq. NH_3 48 hrs. at room temp. in a sealed ampul. Heating V (n = 4) with an equal vol. of liquid NH_3 8–10 hrs. [illegible] gives [illegible] 86% yield by [illegible] 120–40°. V may [illegible] be converted to III having an even no. of C atoms ([illegible]). [illegible] NH_3 acids are obtained from IV via 1,1-dichloroalkanes (C.A. 49, 22[illegible]) and the corresponding [illegible] (C.A. 50, 99985). Similarly, II (n = 2) is converted to $Cl(CH_2)_3CH:CCl_2$ (VI), treated with Cl in H_2SO_4 to give 1,6-dichlorohexanoic acid, which gives 50% DL-proline. Alternatively, VI may be converted via 1,1-

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Nesmeyanov, A.N.; Steepikheev, A.A.; ...

dichloro-5-phthalimido-1-pentene to *3-chloro-5-phthalimidovaleric acid*, from which DL-*ornithine hydrochloride* is obtained in 30% yield, via *2-amino-5-phthalimidovaleric acid*. II are sources of α,ω-dicarboxylic acids via ω,ω,ω-trichloronitriles or via V. II (n = 2) is heated 3 hrs. at 100° with NaCN in $MeOCH_2CH_2OH$ or $EtOCH_2CH_2OH$ to give 83-5% 6,6,6-*trichlorocapronitrile* which, heated 1-2 hrs. in 80-5% H_2SO_4 at 100-25°, [illegible] (VIII) is obtained [illegible] yield from II (n = 3), but prepd. of sebacic acid from II (n = 4) is unsatisfactory. Side-reactions lower yields of VII and VIII via V, but the Na salt of V (n = 8) gives 50-80% *sebacic half nitrile* on treatment with aq. NaCN. Treatment of V (n = 4, 6, or 8) with aq. Na_2CO_3 or NaOH at 140-50° gives the corresponding ω-OH acids, which may be oxidized with concd. HNO_3 to *glutaric*, *pimelic*, and *azelaic acids*, resp., in 70-80% yield. By a process involving a coupling dimerization (*C.A.* 50, 7001*f*), II (n = 2 or 3) serve as sources of *dodecanedioic* and *thapsic* acids, resp. II or V react with Na_2S to give $S[(CH_2CH_2)_nCCl_3]_2$ (IX) or $S[(CH_2CH_2)_nCO_2H]_2$ (X), or IX may be converted to X by treatment with concd. H_2SO_4 (*C.A.* 49, 6120*g*), esters of X being valuable as plasticizers. IX (n = 2), m. 86°, is obtained in 88% yield from II (n = 2). X (n = 2, 80% from V, n = 4), m. 94-5° (from H_2O). *sulfoxide* (prepd. by treating X with 1 equiv. 30% H_2O_2 with cooling, yield 70%), m. 88-9° (from acetone); *sulfone* (prepd. by warming X with excess 30% H_2O_2, 90% yield), m. 180-1° (from EtOH); *Et ester*, b_{10} 160-70°, n_D^{20} 1.4665, d_{20} 1.0315; *Bu ester*, b_1 194-5°, n_D^{20} 1.4670, d_{20} 0.9936; *hexyl ester*, b_1 237-0°, n_D^{20} 1.4670, d_{20} 0.9688; *heptyl ester*, b_1 240-0°, n_D^{20} 1.4665, d_{20} 0.9574; [illegible] d_{20} 0.9463. X [illegible] prepd. in [illegible] yield from V (n = 4) m. 96-7° (fr[illegible] St-

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Nesmeyanov, A. N.; Stepanishchev, A. I.

OH); *Et ester*, b. 183-4°, n_D 1.4608, d_4 0.9951; *Bu ester*, b. 214-15° (n_D 1.4641, d_4 0.9878). X (n = 4) (92% from V, n = 8) m. 99-9° (from EtOH); *sulfone* (90%), m. 145-9° (from EtOH); *Et ester*, b. 210-20°, m. 87-8° (from petr. ether); *Bu ester* b. 230-40°, m. 21-3° (from petr. ether). Polymerization [illegible] of III [illegible] 3), studied [illegible] ("Onanth") [illegible] monomer); hot H_2O exts. [illegible] fractional recrystn. from H_2O and dioxane, cryoscopy in PhOH, and the absence of free CO_2H and NH_2 to consist of cyclic dimers and trimers. III (n = 3) is similarly completely polymerized. Onanth (XI), m. 225°, is sol. only in concd. mineral acids, PhOH, and [illegible]OH, has a hygroscopicity of 2.2-2.4% (at a relative humidity [illegible]%), is resistant to common solvents, and is less [illegible] by mineral acids than is the analogous resin Kapron (the German Perlon). The viscosity of XI is stabilized by 0.1-2.6 mole-% of HOAc, NaOH, or VII. XI is a heat-stable fiber for 16 hrs. at 230°. The physico-mech. properties of its fibers compare favorably with those of Kapron and of Nylon; like those of Kapron, their tensile strength and stretchability change little with moisture. Exposed to ultraviolet light (from a PRK-2 lamp) for 24 hrs. at 150°, fibers of Onanth suffered a loss of 15-17% of strength, while those of Kapron lost 45-50%. Studies of the effects of temp. on the strength and stretchability of Onanth reveal considerable crystallinity. Polymerization of III (n = 4) yields a white resin (Pelargon) (XII), m. 200-2°, less sol. and less hygroscopic (1.2% at a relative humidity of 65%) than XI, plastic (can be cut with a knife), and amenable to cold-drawing. XII has high heat-stability at 200-300°. XI-XII copolymers are prepd. (by heating 99:1 to 1:95 mixts. 1 hr. at 200° in N-filled ampuls) and studied. Up to 5% XII in XI has no appreciable effect on its properties; larger amts. of XII depress the m.p. of the resin, reaching a min. (approx. 157°) at 60% XII; the 40:60, 50:50, and 60:40 copolymers are sol. in hot 90% aq. EtOH or H_2O.

David R. Howton

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FREYDLINA, R. KH.

AUTHOR: Freydlina, R. Kh. 62-11-6/29

TITLE: Telomerization Reaction and Chemical Transformation of Telomers (Reaktsiya telomerizatsii i khimicheskiye prevrashcheniya telomerov).

PERIODICAL: Izvestiya AN SSSR, Otdel. Khim. Nauk, 1957, Nr 11, pp. 1333-1343 (USSR)

ABSTRACT: Out of the aliphatic compounds for a synthesis at present the low-molecular substances with 1-6 carbon atoms in the molecule on the one hand, and the high-molecular compounds with many thousands of atoms in the molecule on the other hand are the most accessible ones. In between them is the area of the compounds with "average" molecular weights. The opinion is represented that the telomerization-reaction, as a reaction of controlled polymerization, will offer the possibility 1.) to penetrate into this field little investigated, 2.) to construct easily molecules with given final groups and 3.) to obtain a set of individual telomer-homologues by means of this reaction and thereby to produce series of matters with the same structure. The investigation carried out here shows great possibilities

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FREYDLINA, R. KH.

AUTHOR: Freydlina, R. Kh., Doctor of Chemical Sciences 30-11-7/23

TITLE: The Use of the Telomerization-Reaction in the Organic Synthesis (Primeneniye reaktsii telomerizatsii v organicheskom sinteze)

PERIODICAL: Vestnik AN SSSR, 1957, Vol. 27, Nr 11, pp. 69-77 (USSR)

ABSTRACT: One of the most promising possibilities of using natural and industrial gases for heavy organic syntheses is the telomerization-reaction by means of which (starting from simplest olefins) various important mono-, bi- and poly-functional compounds can be obtained. (See formulae (1) and (2)). The reaction of the telomerization of ethylene and carbon tetrachloride on an experimental scale was for the first time realized in the USSR. Then the author deals with the thermal telomerization of olefins with silanes (containing the binding Si - H). Only one example of telomerization in this field was hitherto described in publications (interaction of perfluoroethylene with 3-chlorosilane under the influence of ultraviolet light). In the Institute for Elementary-Organic Compounds AN USSR, however, the reaction of the thermal telomerization of olefins with silanes was found (see table 1). After the reaction of the telomerization between ethylene and carbon tetrachloride, described by the chemists R.Dzhoys, N.Khanford and I.Kharmond, the formerly unknown $\alpha,\alpha,\alpha,\omega$-tetrachloro-

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The Use of the Telomerization-Reaction in the Organic Synthesis. 30-11-7/23

alkanes became easily accessible substances. Then the author gives a description of the synthesis on the basis of $\alpha,\alpha,\alpha,\omega$-tetrachlorsilanes. Finally the author treats the production of synthesis substances, starting from the $\alpha,\alpha,\alpha,\omega$-tetrachloroalkanes of the telomerization of ethylene with carbon tetrachloride. There are 1 table, and 23 references, 21 of which are Slavic

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