

spectra of compounds **IV**, **V** the absorption bands of C=O bond vibrations appear respectively at 1721 and 1744 cm^{-1} . To the OH group of acid **IV** corresponds a broad band in the region 2500–3200 cm^{-1} , to the vibrations of C–Cl bonds in both compounds belong the absorption bands in the 736–823 cm^{-1} range. In the ^1H NMR spectrum of acid **I** the signals of CH_2 and COOH groups were found as a singlet at 3.72 ppm and a broadened singlet at 10.10 ppm respectively. In the ^1H NMR spectrum of ester **V** the signal of methoxy group appears as a singlet at 3.74 ppm, and a singlet of methylene group is observed at 3.54 ppm. In the mass spectrum of ester **V** in the group of molecular ion peaks the ratio of intensities of the main isotope components (61:100:65:21) corresponds to the presence of five chlorine atoms in the molecule [4, 5].

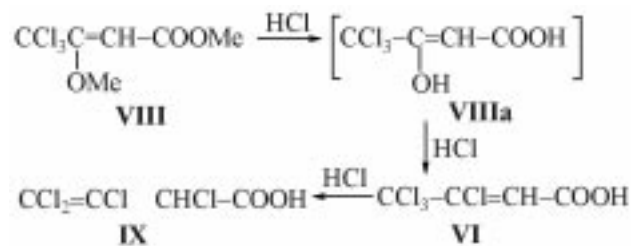
The dehydrochlorination of acid **IV** and ester **V** was studied under various conditions. It was found that dehydrochlorination with aqueous KOH in the presence of triethylbenzylammonium chloride as phase-transfer catalyst and also dehydrochlorination with triethylamine in methanol is accompanied by tarring, and the yield of target products does not exceed 5–15%. Reactions with water solutions of sodium hydrogen carbonate and potassium carbonate failed to occur. The optimum procedure consisted in the use of pyridine in methanol solution at heating to 60°C. Under these conditions acid **IV** underwent more selective dehydrochlorination furnishing in 55% yield 3,4,4,4-tetrachloro-2-butenoic acid (**VI**). The dehydrochlorination of compound **V** gave rise to methyl 3,4,4,4-tetrachloro-2-butenoate (**VII**) in 38% yield. In the reactions an appreciable amount of tar was formed.

The composition and structure of products **VI** and **VII** were confirmed by elemental analysis and IR, ^1H , and mass spectra, and also by comparison of the physical characteristics with the published data [6]. The presence of the central C=C bond is proved by the appearance in the IR spectra of compounds **VI**, **VII** of a characteristic absorption band at 1629 and 1634 cm^{-1} respectively. In the ^{13}C NMR spectra the signals of trichloromethyl groups are observed at δ 95.29 ppm for acid **VI** and 95.28 ppm for ester **VII**. The appearance of signals from carbons in =C³Cl- moiety in the ^{13}C NMR spectra of compounds **VI** and **VII** at 147.36 and 144.82 ppm respectively evidence that both the acid **VI** and ester **VII** form as *Z*-isomers: Preliminary detailed studies of ^{13}C NMR spectra from a series of chlorobutenoic acids and their esters has demonstrated that the signals of carbon atoms of =C³Cl- moieties in the *E*-isomers of

chlorinated crotonic acids appear in stronger field, in 130 ppm region, whereas the corresponding signals for the *Z*-isomers are present at 140–148 ppm [7].

The treatment of ester **VII** with sodium methylate solution in methanol provided in 60% yield a product of internal chlorine substitution by a methoxy group, the corresponding methyl 3-methoxy-4,4,4-trichloro-2-butenoate (**VIII**) whose structure was proved by spectral data (see EXPERIMENTAL). The characteristic signal in the ^{13}C NMR spectrum of compound **VIII** at δ 94.73 ppm belonging to the trichloromethyl group [8] indicated, that the group remained intact, and the substitution occurred at C³ atom.

The hydrolysis of esters **VII**, **VIII** with 20% hydrochloric acid took an uncommon course, and instead of the corresponding crotonic acids gave rise to 2,3,4,4-tetrachloro-3-butenoic acid that formed by isomerization of the 3,4,4,4-tetrachloro-2-butene skeleton. Therewith in ester **VIII** occurred also a formal substitution of chlorine for methoxy group in position 3 of the molecule. Presumably first an enol structure **VIIIa** formed followed by substitution of the hydroxy group by chlorine and rearrangement into 2,3,4,4-tetrachloro-3-butenoic acid (**IX**). By a special experiment we demonstrated that boiling of acid **VI** in 20% hydrochloric acid for 8 h resulted in its complete isomerization into acid **IX**.

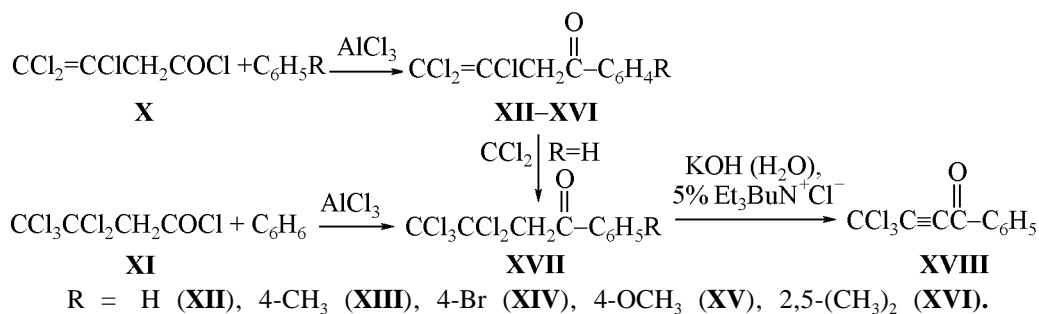


In the IR spectrum of acid **IX** to the vibrations of the terminal C=C bond corresponds an absorption band at 1593 cm^{-1} , to those of C–Cl bond the bands at 752, 848, and 923 cm^{-1} . In the ^1H NMR spectrum the proton of the CHCl group appears as a singlet with the chemical shift of 5.85 ppm, and to the COOH group corresponds a broadened singlet at δ 10.41 ppm. The ^{13}C NMR spectrum of acid **IX** lacks the signals of trichloromethyl group in the region 85–95 ppm and of =CH group that are characteristic of the crotonic acid **VI**; instead signals appear from CHCl group at 57.88 ppm ($J_{\text{C-H}}$ 150.5 Hz) and from carbon atoms of trichlorovinyl moiety at δ 125.72 and 127.02 ppm.

The available trichlorobutenoic (**I**) and pentachlorobutanoic (**IV**) acids can serve as convenient

initial compounds for preparation of chlorine-substituted alkyl aryl ketones. Chlorine-substituted ketones are interesting substances applied to preparation of helminthicides, insecticides, and the other biologically active compounds [9]. Some among them are more active than penicillin and streptomycin.

By treating acids **I**, **IV** with solution of thionyl chloride in carbon tetrachloride the corresponding acyl chlorides **X**, **XI** were obtained in 95 and 74% yield respectively. Then we used compounds **X**, **XI** for acylation of aromatic hydrocarbons along Friedel-Crafts method to prepare ketones **XII-XVII**.



The acylation took 3–4 h and afforded ketones with trichloroallyl (**XII-XVI**) or pentachloropropyl (**XVII**) group and with different substituents in the aromatic ring in 48–75% yields.

The composition and structure of ketones **XII-XVII** obtained were confirmed by elemental analysis and IR, ^1H , and mass spectra. The relevant data are given in a table. In the IR spectra are observed the absorption bands from vibrations of the following bonds: C=O, at $1677\text{--}1700\text{ cm}^{-1}$; C=C, at $1555\text{--}1617\text{ cm}^{-1}$; C-Cl, at $726\text{--}841\text{ cm}^{-1}$. In the ^1H NMR spectra of compounds **XII-XVII** the singlet in the region 4.19–4.28 ppm is assigned to the protons of CH_2 group, and multiplets at δ 6.90–8.00 ppm to the protons of aromatic ring. In the spectra were also observed the signals from the substituents attached to the aromatic ring with an appropriate integral intensity. In the mass spectra of ketones the groups of molecular ion peaks are present where the relative intensity of the main isotope components equal to 100:98:32:3.5 for compounds **XII-XVI** and 61:100:65:21 for compound **XVII** indicating the presence in the molecule of three and five chlorine atoms respectively [4, 5].

The chlorination of 2,3,3-trichloroallyl phenyl ketone (**XII**) with gaseous chlorine in CCl_4 under sunlight was investigated. It was shown that the reaction occurred exclusively at the double bond if the trichlorovinyl group of compound **XII** giving rise to 2,2,3,3,3-pentachloropropyl phenyl ketone (**XVII**) in preparative yield of 92%. The compound was identical by its physical characteristics and spectra to the product of benzene alkylation with acyl chloride **XI**; this fact additionally supports its structure. No

products of hydrogen substitution by chlorine in the methylene group or aromatic ring was detected.

A reaction of dehydrochlorination of 2,2,3,3,3-pentachloropropyl phenyl ketone **XVII** with aqueous alkali was studied under conditions of phase-transfer catalysis. It was established that under optimal conditions [at the molar ratio of reagents ketone:KOH (20% water solution):triethylbenzylammonium chloride equal to 1:4:0.05] the reaction at room temperature took 3 h and resulted in a cleavage of two HCl molecules furnishing 3,3,3-trichloro-1-propynyl phenyl ketone **XVIII** in 46% yield. At reduced relative amount of alkali or at its lower concentration a mixture of products formed, and some of initial ketone **XVII** remained unreacted. The raising of temperature in this case did not provide positive result and favored decomposition of the initial compounds and reaction products.

The structure of 3,3,3-trichloropropynyl phenyl ketone (**XVIII**) was confirmed by elemental analysis, IR, ^1H and ^{13}C NMR spectra, and by GC-MS method. In the IR spectrum of compound **XVIII** appear the absorption bands of carbonyl group vibrations at 1644 cm^{-1} , of C=C bonds from the benzene ring at 1583 and 1598 cm^{-1} , of acetylene C=C bond at 2218 cm^{-1} , of C-Cl bond at 763 cm^{-1} .

In the ^1H NMR spectrum of compound **XVIII** to the protons of aromatic ring belong multiplets with the chemical shifts of 7.63 ppm (3H arom) and 8.09 (2H arom). In the ^{13}C NMR spectrum registered with decoupling from protons appear signals at δ 76.7 and 83.8 ppm characteristic of acetylene carbons, and a signal at δ 86.0 ppm corresponding to the trichloro-

Yields, melting points, IR, ¹H NMR spectra, and elemental analyses of ketones **XII–XVII**

Compd. no.	Yield, %	mp, °C	IR spectrum, ν , cm^{-1}	¹ H NMR spectrum, δ , ppm
XII	75	45–46	1690(C=O), 1584, 1600, 1617(C=C), 749, 784	4.28 s (2H, CH ₂), 7.55 m (3H arom), (C–Cl) 7.95 m (2H arom)
XIII	71	75–76	1683 (C=O), 1568, 1608 (C=C), 758, 823 (C–Cl)	2.42 s (3H, CH ₃), 4.25 s (2H, CH ₂), 7.28 d (2H arom), 7.83 d (2H arom, <i>J</i> 7.7 Hz)
XIV	48	82–83	1686 (C=O), 1571, 1590, 1614 (C=C), 767, 830 (C–Cl)	4.24 s (2H, CH ₂), 7.64 d (2H arom), 7.28 d 7.80 d (2H arom, <i>J</i> 8 Hz)
XV	55	66–67	1679 (C=O), 1592, 1614 (C=C), 766, 785, 841 (C–Cl)	3.85 s (3H, OCH ₃), 4.22 s (2H, CH ₂), 6.93 d (2H arom), 7.91 d (2H arom, <i>J</i> 9 Hz)
XVI	70	48–49	1677 (C=O), 1555, 1571, 1607 (C=C), 761, 830 (C–Cl)	2.36 s (3H, CH ₃), 2.46 s (3H, CH ₃), 4.19 s (2H, CH ₂), 7.13 d and 7.23 d (2H arom, <i>J</i> 8 Hz), 7.41 br.s (1H arom)
XVII	58	91–92	1700 (C=O), 1578, 1595 (C=C), 726, 764, 793 (C–Cl)	4.27 s (2H, CH ₂), 7.55 m (3H arom), 7.98 m (2H arom)

Compd. no.	Found, %			Formula	Calculated, %			<i>M</i>	
	C	H	Cl		C	H	Hlg	found ^a	calc.
XII	48.77	2.80	42.41	C ₁₀ H ₇ Cl ₃ O	48.13	2.83	42.63	248	249.55
XIII	50.46	3.22	40.13	C ₁₁ H ₉ Cl ₃ O	50.13	3.44	40.36	262	263.55
XIV	36.70	2.08	56.43	C ₁₀ H ₆ Cl ₃ BrO	36.57	1.84	56.72	326	328.43
XV	46.89	3.50	38.16	C ₁₁ H ₉ Cl ₃ O ₂	47.26	3.25	38.05	278	279.55
XVI	52.10	4.34	38.59	C ₁₂ H ₁₁ Cl ₃ O	51.92	4.00	38.32	276	277.58
XVII	37.22	2.45	54.51	C ₁₀ H ₇ Cl ₅ O	37.48	2.20	55.32	318	320.44

^a Molecular weight determined from mass spectra (values listed in table are for ions *M*⁺ containing ³⁵Cl isotope).

methyl group [8]. It follows from the spectrum registered in the DEPT mode that to the aromatic ring carbons correspond the signals with the chemical shifts 129.4, (C³H, C⁵H), 130.1 (C²H, C⁶H), 135.6 (C⁴H) and 136.2 (C¹) ppm. The signal from the carbonyl group is located at 175.9 ppm. The mass spectrum of ketone contained the isotope peaks of molecular ion of *m/z* 246 (calculated for ³⁵Cl isotope).

The ketones obtained are convenient synthons for further application to the organic synthesis of poly-functional compounds.

EXPERIMENTAL

IR spectra were recorded on Fourier spectrophotometer Protege-460 from KBr pellets (compounds **IV–VI**, **IX**, **XI–XVIII**) or from thin film (compounds **VII**, **VIII**, **X**, **XI**). NMR spectra were registered on spectrometer Tesla -567A (100 MHz) in CDCl₃ solutions, chemical shifts measured relative

to TMS. Mass spectra were measured on mass spectrometer MKh-1320 at ionizing electrons energy 50 eV. GC-MS analysis was performed on Hewlett-Packard GC/MS 5890/5972 instrument.

The reaction progress was monitored and the products were analyzed by GLC on chromatograph Chrom-41 equipped with glass columns 1500 × 3 mm, stationary phase 5% SE-30 on Chromaton N-AW-DMCS (0.160–0.200 μ). detector katharometer, oven temperature 160°C, carrier gas helium.

3,4,4-Trichloro-3-butenic acid (**I**) was prepared by procedure [3] from 2-H-pentachloro-1,3-butadiene (**II**).

3,3,4,4,4-Pentachlorobutanoic acid (IV). Through a solution of 5.7 g (0.03 mol) of 3,4,4-trichloro-3-butenic acid (**I**) in 25 ml of carbon tetrachloride under sunlight was passed a stream of chlorine for 30 min. The separated colorless crystal-

line precipitate was filtered off and washed with cold CCl_4 . We obtained 7.05 g (90%) of 3,3,4,4,4-pentachlorobutanoic acid **IV**, mp 132–134°C (from CCl_4). Found, %: C 18.70; H 1.41; Cl 67.88. M_{eq} 260. $\text{C}_4\text{H}_3\text{Cl}_5\text{O}_2$. Calculated, %: C 18.45; H 1.16; Cl 68.09. M 260.34.

Methyl 3,3,4,4,4-pentachlorobutanoate (V). In 30 ml of anhydrous methanol was dissolved 1.3 g (5 mmol) of acid **IV**, 3 drops of concn. H_2SO_4 was added, and the mixture was boiled for 8 h. Then the mixture was poured into water, the precipitate was filtered off, washed with water, dried in a vacuum, and recrystallized from hexane. We obtained 1.26 g (92%) of ester **V**, mp 50–51°C. Found, %: C 22.29; H 2.15; Cl 64.20. $[M]^+$ 272 (for ^{35}Cl). $\text{C}_5\text{H}_5\text{Cl}_5\text{O}_2$. Calculated, %: C 21.89; H 1.84; Cl 64.61. M 274.37.

Dehydrochlorination of 3,3,4,4,4-pentachlorobutanoic acid. A mixture of 5 g (19.2 mmol) of 3,3,4,4,4-pentachlorobutanoic acid, 4.56 g (57.6 mmol) of pyridine, and 15 ml of methanol was stirred for 8 h at 60°C (GLC monitoring), then the mixture was cooled and poured into cold water, acidified with HCl to pH 3, and the reaction product was extracted with dichloromethane. The extract was dried with CaCl_2 , the solvent was removed, and the solid residue was recrystallized from hexane. We obtained 2.37 g (55%) of Z-3,4,4,4-tetrachloro-2-butenic acid (**VI**), mp 83–85°C in agreement with the published data [6]. IR spectrum, cm^{-1} : 781, 817 (C–Cl), 1620 (C=C), 1699 (C=O), 3083 (C–H). ^1H NMR spectrum, δ , ppm: 7.13 s (1H, =CH), 10.78 br.s (1H, COOH). ^{13}C NMR spectrum, δ , ppm: 95.29 (CCl_3), 119.15 (=CH, $J_{\text{C-H}}$ 168.1 Hz), 147.36 (=CCl), 168.27 (C=O). Found, %: C 21.59; H 1.09; Cl 63.01. M_{eq} 222. $\text{C}_4\text{H}_2\text{Cl}_4\text{O}_2$. Calculated, %: C 21.46; H 0.90; Cl 63.35. M 223.87.

Dehydrochlorination of methyl 3,3,4,4,4-pentachlorobutanoate (V) was carried out in a similar way, but the product was purified by vacuum distillation. Yield of methyl 3,4,4,4-tetrachloro-2-butenate (**VII**) 38%, bp 50–52°C (~1 mm Hg). d_4^{20} 1.523. n_D^{20} 1.5120. IR spectrum, cm^{-1} : 781, 821 (C–Cl), 1634 (C=C), 1741 (C=O), 3084 (C–H). ^1H NMR spectrum, δ , ppm: 3.83 s (3H, OCH_3), 7.06 s (1H, =CH). ^{13}C NMR spectrum, δ , ppm: 52.87 (OCH_3 , $J_{\text{C-H}}$ 147.7 Hz), 95.28 (CCl_3), 119.67 (=CH, $J_{\text{C-H}}$ 167.4 Hz), 144.82 (=CCl), 163.12 (C=O). Found, %: C 24.89; H 1.39; Cl 60.01. $[M]^+$ 236 (for ^{35}Cl). $\text{C}_5\text{H}_4\text{Cl}_4\text{O}_2$. Calculated, %: C 25.24; H 1.69; Cl 59.61. M 237.90.

Methyl 3-methoxy-4,4,4-trichloro-2-butenate (VIII). To a solution of 2 g (8.4 mmol) of methyl

3,4,4,4-tetrachloro-2-butenate (**VII**) in 5 ml of MeOH at 25°C while vigorous stirring was added dropwise 0.68 g of sodium methylete in 5 ml of methanol. The mixture was stirred for 2 h, the precipitate was filtered off, the filtrate was poured into water, and the product was extracted with ether. The extract was dried with CaCl_2 , the solvent was removed. The vacuum distillation of the residue afforded 1.18 g (60%) of ester **VIII**. bp 80–81°C (~4 mm Hg). d_4^{20} 1.427. n_D^{20} 1.5031. IR spectrum, cm^{-1} : 780, 827 (C–Cl), 1646 (C=C), 1730 (C=O), 3088 (=C–H). ^1H NMR spectrum, δ , ppm: 3.78 s (3H, COOCH_3), 4.12 s (3H, OCH_3), 6.28 s (1H, =CH). ^{13}C NMR spectrum, δ , ppm: 52.34 (OCH_3 , $J_{\text{C-H}}$ 147.6 Hz), 65.67 (OCH_3 , $J_{\text{C-H}}$ 147.8 Hz), 94.73 (CCl_3), 101.20 (=CH, $J_{\text{C-H}}$ 165.5 Hz), 164.82 and 164.83 (=CCl and C=O). Found, %: C 30.46; H 3.14; Cl 45.93. $[M]^+$ 232 (for ^{35}Cl). $\text{C}_6\text{H}_7\text{Cl}_3\text{O}_3$. Calculated, %: C 30.87; H 3.02; Cl 45.55. M 233.48.

Hydrolysis of methyl 3,4,4,4-tetrachloro-2-butenate (VII). A mixture of 3.05 g (12.8 mmol) of ester **VII** and 30 ml of 20% aqueous HCl was stirred at 100°C for 10 h. On cooling the mixture to 25°C the reaction product was extracted with dichloromethane, extract was dried on CaCl_2 , the solvent was removed, and by vacuum distillation was isolated 1.15 g (40%) of 2,3,4,4-tetrachloro-3-butenic acid (**IX**), mp 80–81°C [11]. ^{13}C NMR spectrum, δ , ppm: 57.88 (CHCl , $J_{\text{C-H}}$ 150.5 Hz), 125.72 and 127.02 ($\text{CCl}_2=\text{CCl}-$), 170.02 (C=O). Found, %: C 21.72; H 1.21; Cl 63.44. M_{eq} 220. $\text{C}_4\text{H}_2\text{Cl}_4\text{O}_2$. Calculated, %: C 21.46; H 0.90; Cl 63.35. M 223.87.

Hydrolysis of methyl 3-methoxy-4,4,4-trichloro-2-butenate (VIII) was carried out similarly, but the reaction took 3 h.

3,4,4-Trichloro-3-butenoyl chloride (X). To a solution of 2.86 g (15.1 mmol) of 3,4,4-trichloro-3-butenic acid (**I**) in 10 ml of CCl_4 was added dropwise 3.3 ml (45.4 mmol) of thionyl chloride, the reaction mixture was stirred for 30 min and boiled to the end of HCl liberation (monitoring with litmus paper and GLC). Then the solvent and excess thionyl chloride were distilled off, and the residue was distilled in a vacuum. We obtained 2.99 g (95%) of acyl chloride **X**, bp 38–39°C (1 mm Hg). d_4^{20} 1.558. n_D^{20} 1.5135. IR spectrum, cm^{-1} : 761, 792 (C–Cl), 1607 (C=C), 1800 (C=O). ^1H NMR spectrum, δ , ppm: 4.12 s (2H, CH_2). Found, %: C 23.02; H 1.37; Cl 68.55. $[M]^+$ 206 (for ^{35}Cl). $\text{C}_4\text{H}_2\text{Cl}_4\text{O}$. Calculated, %: C 23.11; H 0.97; Cl 68.22. M 207.87.

3,3,4,4,4-Pentachlorobutanoyl chloride (XI) was obtained in a similar way from acid **VI** and thionyl chloride. Yield 74%, bp 67–68°C (2 mm Hg). d_4^{20} 1.673. n_D^{20} 1.5308. IR spectrum, cm^{-1} : 740, 788, 823 (C–Cl), 1827 (C=O). ^1H NMR spectrum, δ , ppm: 4.16 s (2H, CH_2). Found, %: C 17.01; H 0.59; Cl 76.28. $[M]^+$ 276 (for ^{35}Cl). $\text{C}_4\text{H}_2\text{Cl}_6\text{O}$. Calculated, %: C 17.23; H 0.72; Cl 76.30. M 278.78.

2,3,3-Trichloroallyl phenyl ketone (XII). To 2.26 g (10.88 mmol) of 3,4,4-trichloro-3-butenoyl chloride (**X**) at 10–12°C was added 1.46 g (10.94 mmol) of anhydrous AlCl_3 , and the reaction mixture was stirred for 20 min. Then to the mixture was added 1.02 g (13.06 mmol) of benzene, and the stirring was continued for 3 h at room temperature and then for 2 h at 60°C. After completion of the reaction (GLC monitoring) the reaction mixture was diluted with carbon tetrachloride (1:1) and poured on ice. The organic layer was separated, washed with water, with diluted solution of potassium carbonate, again with water, and dried with magnesium sulfate. The solvent was distilled off in a vacuum, and the solid residue was recrystallized from ethanol. We obtained 1.90 g (70%) of trichloroallyl phenyl ketone (**XII**), mp 45–46°C.

Ketones **XIII–XVI** were obtained by a similar procedure from acyl chloride **X** and an appropriate substituted benzene. The synthesis of 2,2,3,3,3-pentachloropropyl phenyl ketone **XVII** was carried out likewise from pentachlorobutanoyl chloride **XI** and benzene. The yields and physical characteristics of compounds obtained are presented in the table.

Chlorination of 2,3,3-trichloroallyl phenyl ketone (XII). Through a solution of 1.77 g (7.1 mmol) of 2,3,3-trichloroallyl phenyl ketone (**XII**) in 10 ml of benzene was passed a stream of chlorine for 4 h (GLC monitoring). Then the solvent was removed, and the residue was recrystallized from ethanol. We obtained 2.09 g (02%) of ketone **XVII**. Dehydrochlorination of 2,2,3,3,3-pentachloropropyl phenyl ketone (**XVII**). To 0.16 g (0.7 mmol) of triethylbenzylammonium chloride in 30 ml of 20% water solution of KOH at vigorous stirring was added a solution of 1 g (3.12 mmol) of 2,2,3,3,3-pentachloropropyl phenyl ketone **XVII** in 5 ml of ethyl ether. The stirring continued for 3 h. Then the organic layer was separated, the solvent was removed, and the

product obtained was recrystallized from hexane. We obtained 0.35 g (46%) of 3,3,3-trichloro-1-propynyl phenyl ketone (**XVIII**), mp 84–86°C. Found, %: C 48.68; H 1.87; Cl 42.58. $[M]^+$ 246 (for ^{35}Cl). $\text{C}_{10}\text{H}_5\text{Cl}_3\text{O}$. Calculated, %: C 48.53; H 2.04; Cl 42.97. M 247.51.

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