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Unusual Reaction of Chloroacetyl Chloride with 1,2-Dichloroethene. Synthesis and Properties of 2-Chlorovinyl Dichloromethyl Ketone

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Abstract—The reaction of chloroacetyl chloride with 1,2-dichloroethene in the presence of $AlCl_3$ unexpectedly led to the formation of (*E*)-1,1,4-trichlorobut-3-en-2-one whose structure was proved by ^1H and ^{13}C NMR, IR, and mass spectra and independent synthesis. A probable reaction scheme was proposed, which involves transformation of initially formed 1,2,4-trichloro-3-oxobutan-2-yl cation by the action of $AlCl_3$. The high reactivity of the vinylic halogen atom in (*E*)-1,1,4-trichlorobut-3-en-2-one was demonstrated by its reactions with nitrogen-centered nucleophiles (triethylamine, aniline, 3,5-dimethyl-1*H*-pyrazole) and sodium sulfide. These reactions involved only the C–Cl bond in the vinyl fragment and afforded (4,4-dichloro-3-oxobut-2-en-1-yl)triethylammonium chloride, 1,1-dichloro-4-phenylaminobut-3-en-2-one, 1-(4,4-dichloro-3-oxobut-2-en-1-yl)-3,5-dimethyl-1*H*-pyrazole, and 4,4′-thiobis(1,1-dichlorobut-3-en-2-one), respectively. The reaction of 1,1,4-trichlorobut-3-en-2-one with benzylhydrazine gave a mixture of 1,3- and 1,5-disubstituted pyrazoles.

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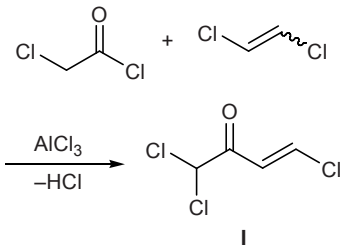
Chlorovinyl ketones are very important and promising synthons for the preparation of a large number of highly reactive polyfunctional heterocyclic compounds [1]. We previously synthesized alkyl 1,2-dichlorovinyl ketones from aliphatic carboxylic acid chlorides and 1,2-dichloroethylene in the presence of aluminum chloride, and the product structure was studied by ^1H and ^{13}C NMR, IR spectroscopy, and quantum-chemical calculations [2]. We also reported [2] that neither aryl nor trichloromethyl 1,2-dichlorovinyl ketones can be obtained in such a way. Therefore, it seemed important to determine the scope of application of the above synthetic approach to 1,2-dichlorovinyl ketones and examine the chemical properties of new halo enones.

In the present work we studied the reaction of chloroacetyl chloride with 1,2-dichloroethylene. Prior to our study, only one chloroalkyl 1,2-dichlorovinyl ketone has been reported, namely dichloromethyl 1,2-dichlorovinyl ketone which was synthesized from dichloroacetyl chloride and *cis*-1,2-dichloroethylene in the presence of aluminum chloride at 35°C (reaction time 40 h) [3]. It was also found [2, 3] that reactions of aliphatic carboxylic acid chlorides and dichloroacetyl chloride with 1,2-dichloroethylene give rise to mixtures of the corresponding 1,2,2-trichloroethyl alkyl (or dichloromethyl) ketones and their thermal dehydrochlorination products, 1,2-dichlorovinyl alkyl (or dichloromethyl) ketones. Complete dehydrochlorination of 1,2,2-trichloroethyl ketones was accomplished only by prolonged heating with boiling aqueous sodium carbonate [2]. Treatment of the reaction mixtures with bases at room temperature or steam distillation (as in the procedures for the preparation of aliphatic 2-chloro- and 2,2-dichlorovinyl ketones from 2,2-dichloro- and 2,2,2-trichloroethyl ketones [1, 4]) did not ensure complete conversion of trichloroethyl alkyl (or dichloromethyl) ketones into 1,2-dichlorovinyl alkyl ketones.

We failed to obtain chloromethyl 1,2-dichloroethyl ketone under the conditions analogous to the synthesis of dichloromethyl 1,2-dichlorovinyl ketone [3] and alkyl 1,2-dichlorovinyl ketones [2] from 1,2-dichloroethylene and carboxylic acid chlorides.

Unexpectedly, the reaction of chloroacetyl chloride with commercially available 1,2-dichloroethene (a mixture of *cis* and *trans* isomers) in the presence of aluminum chloride at a reactant ratio of 1:10:1 on heating at 60°C led to the formation of previously unknown 1,1,4-trichlorobut-3-en-2-one in up to 75% yield (Scheme 1).

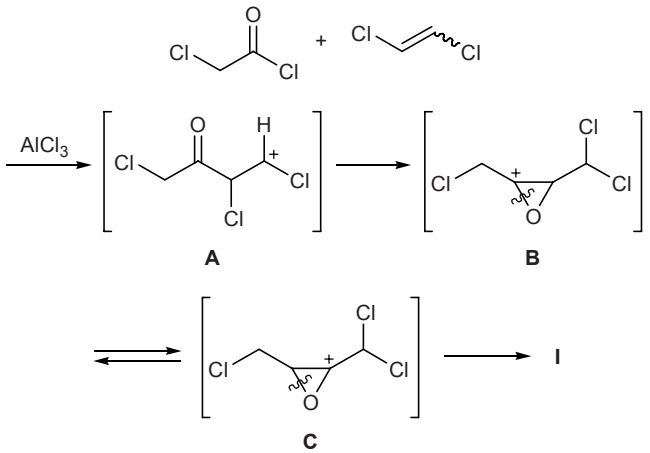
Scheme 1.



I

Taking into account that distillation of the reaction mixture gave only unsaturated ketone **I** and that neither expected saturated chloromethyl 1,2,2-trichloroethyl ketone nor its dehydrochlorination product, chloromethyl 1,2-dichlorovinyl ketone, was detected (even in trace amounts), the mechanism of formation of vinyl ketone **I** attracts specific interest. We previously [5] synthesized unsaturated 2,2-dichlorovinyl trifluoromethyl ketone by reaction of trifluoroacetyl chloride with 1,1-dichloroethene in the presence of $AlCl_3$ at –50 to –60°C using equimolar amounts of the reactants and catalyst. Presumably, intermediate carbocation formed in this reaction was stabilized via elimination of proton rather than by addition of chloride ion to give trichloroethyl trifluoromethyl ketone. The above data suggest different paths of formation of 1,2- and 2,2-dichlorovinyl alkyl ketones and dichloromethyl 1,2-dichlorovinyl ketone [1–3], on the one hand, and ketone **I**, on the other, from acyl chlorides and 1,2- and 1,1-dichloroethene.

Scheme 2.

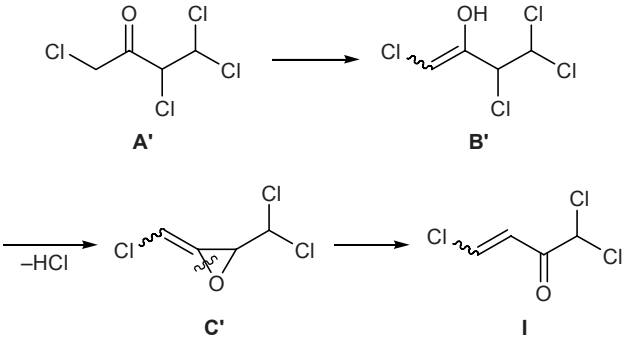


A **B** **C**

By special experiment we showed that 1,2-dichloroethene on heating with $AlCl_3$ on exposure to air is not converted into 1,1,4-trichlorobut-3-en-2-one. Probably, addition of chloroacetyl cation to 1,2-dichloroethene in the presence of aluminum chloride gives carbocation **A** which undergoes isomerization into oxiranyl cation **B** with simultaneous 1,2-chlorotropic rearrangement; the subsequent prototropic rearrangement yields cation **C**. Opening of the oxirane ring in **B** or **C**, followed by stabilization via elimination of proton leads to final product **I** (Scheme 2). In both cases (cations **B** and **C**) cleavage of the oxirane ring occurs at the C–O bond contiguous to the chloromethyl rather than dichloromethyl group. While studying thermal and catalytic decomposition of epoxides derived from chloroalkenes, it was shown [6, 7] that just the C–O bond neighboring to the less electronegative substituent is cleaved.

Analogous transformations of initially formed saturated 1,2,2-trichloroethyl chloromethyl ketone **A′** could also lead to compound **I**. As shown in Scheme 3, the transformation sequence includes enolization of ketone **A′**, dehydrochlorination of enol **B′** to oxirane **C′**, opening of the oxirane ring at the side of the chloromethylidene group, and prototropic rearrangement to give the final product, ketone **I**.

Scheme 3.



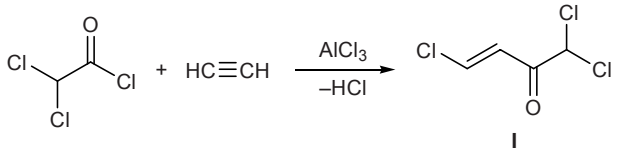
A′ **B′** **C′** **I**

However, this path seems to be less probable. As we found previously [5], alkyl 1,2,2-trichloroethyl ketones are not converted into chlorovinyl chloroalkyl ketones, and they do not undergo dehydrochlorination under the given conditions. No dichloromethyl 2,2-dichlorovinyl ketone was formed in the reaction of dichloroacetyl chloride with 1,2-dichloroethene [3].

We also synthesized 1,1,4-trichlorobut-3-en-2-one (**I**) by independent method, addition of dichloroacetyl

chloride to acetylene in the presence of anhydrous $AlCl_3$ (Scheme 4); however, the yield of **I** in this reaction was lower (56%).

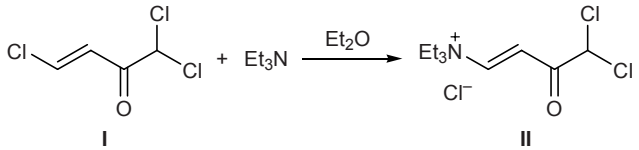
Scheme 4.



The structure of 1,1,4-trichlorobut-3-en-2-one was proved by IR spectroscopy, ^1H and ^{13}C NMR (including two-dimensional ^1H–^{13}C HSQC correlation technique), mass spectrometry, and elemental analysis. The IR spectrum of **I** contained strong absorption bands due to stretching vibrations of the carbonyl group and double C=C bond. In the ^1H NMR spectrum of **I** we observed two doublets from olefinic protons with a coupling constant typical of *trans* orientation of these protons. Using two-dimensional ^1H–^{13}C NMR spectroscopy, we succeeded in assigning signals from protons and carbon atoms in the vinyl group. Thus the chemical shift of the α-carbon atom (with respect to the carbonyl group) is δ_C 124.23 ppm, and that of the β-carbon atom, 141.17 ppm. Ketone **I** showed in the mass spectrum the molecular ion peak with *m*/*z* 173.

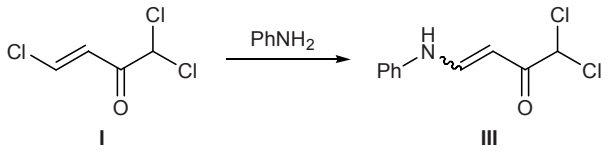
The chlorine atom in position *2* of the vinyl group in molecule **I** is highly reactive. The reaction of ketone **I** with triethylamine was accompanied by heat evolution, and the product was (4,4-dichloro-3-oxobut-2-en-1-yl)triethylammonium chloride (**II**) which was formed in quantitative yield (Scheme 5). Compound **II** is promising as a reagent for the introduction of a 4,4-dichloro-3-oxobut-2-en-1-yl group into organic compounds. It was isolated as a hygroscopic crystalline substance, which is readily soluble in water and alcohol. Its IR spectrum contained absorption bands corresponding to vibrations of Et_3N^+, =C–H, and C=O groups.

Scheme 5.



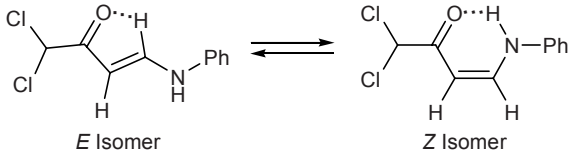
Replacement of the chlorine atom also occurred in the reaction of ketone **I** with aniline, but the resulting 1,1-dichloro-4-phenylaminobut-3-en-2-one (**III**) did not form hydrochloride (Scheme 6). Compound **III** is

Scheme 6.



a crystalline substance readily soluble in alcohols. It displayed in the IR spectrum absorption bands due to vibrations of N–H, C–H, and C=O bonds. Analysis of the ^1H NMR spectra of **III** in DMSO-d_6 showed that it exists as a mixture of *E* and *Z* isomers at a ratio of 2.5 : 1 (Scheme 7). Olefinic protons in the α- and β-positions with respect to the carbonyl group in the *E* isomer resonated at δ 5.81 and 8.21 ppm, respectively, with a coupling constant ^3J of 12.3 Hz, while the corresponding signals of the *Z* isomer were observed at δ 5.57 and 8.01 ppm (J = 7.7 Hz).

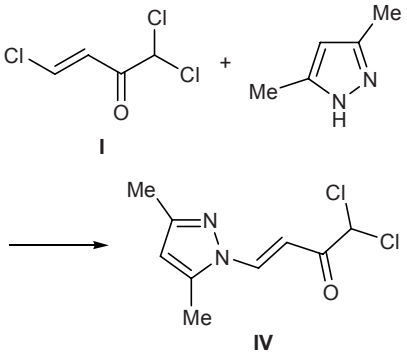
Scheme 7.



Despite variation of the reaction conditions, we failed to obtain product of aniline addition at the carbonyl group, though aromatic amines are known to react with 2-chloro- and 2,2-dichlorovinyl alkyl ketones at both halogen atoms and carbonyl group, yielding phenylimino-2-arylamino- and 2,2-bis(arylamino)vinyl ketones [1, 8, 9].

Chlorovinyl ketone **I** reacted with 3,5-dimethyl-1*H*-pyrazole on heating in alcohol to give 68% of 1-[(*E*)-4,4-dichloro-3-oxobut-1-en-1-yl]-3,5-dimethyl-1*H*-pyrazole (**IV**) (Scheme 8). The IR spectrum of compound **IV** contained absorption bands belonging to vibrations of =C–H bonds, alkyl groups, and carbonyl

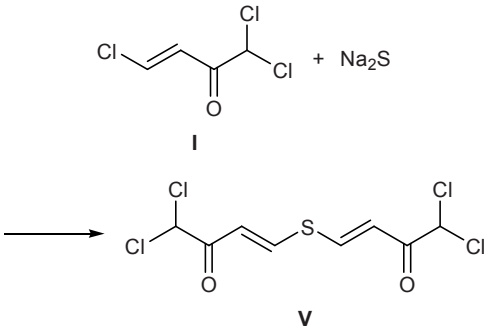
Scheme 8.



group, while spin–spin coupling between the side-chain olefinic protons was characterized by a constant ^3J of 13.3 Hz in the ^{1}H NMR spectrum.

Treatment of ketone **I** with sodium sulfide resulted in replacement of the vinylic chlorine atom by sulfur with formation of 90% of sulfide **V** exclusively as the *E* isomer (Scheme 9). Compound **V** is a solid with a specific odor; it showed in the IR spectrum absorption bands due to =C–H bonds and $CHCl_2$ and C=O groups.

Scheme 9.



The reaction of ketone **I** with benzylhydrazine gave a mixture of regioisomeric 1-benzyl-3-dichloromethyl- and 1-benzyl-5-dichloromethyl-1*H*-pyrazoles **VI** and **VII** at a ratio of ~2:1 (according to the ^{1}H NMR data; Scheme 10). Presumably, the reaction takes two different paths. The first of these involves heterocyclization of the initially formed 2-chlorovinyl ketone hydrazone; as a result, pyrazole **VI** is formed. Following the second path, the primary product is the corresponding 1-benzyl-2-(2-acylvinyl)hydrazine whose heterocyclization yields 1-benzyl-5-dichloromethyl-1*H*-pyrazole (**VII**). In this case, intermediate 4-benzylhydrazino-1,1-dichlorobut-3-en-2-one may be formed via addition of benzylhydrazine to 1,1-dichlorobut-3-yn-2-one arising from dehydrochlorination of initial 2-chlorovinyl ketone. Furthermore, nucleophilic replacement of the halogen atom in **I** and addition to acetylenic ketone may occur with participation of the corresponding hydrazine hydrochloride, so that more nucleophilic NH_2 group rather than $^+NH_2R\ Cl^-$ will be involved. On the other hand, 4-benzylhydrazino-1,1-dichlorobut-3-en-2-one can be formed as a result of intramolecular rearrangement of intermediate 1-benzyl-2-(1,1,4-trichloro-2-hydroxybut-3-en-2-yl)hydrazine (Scheme 11).

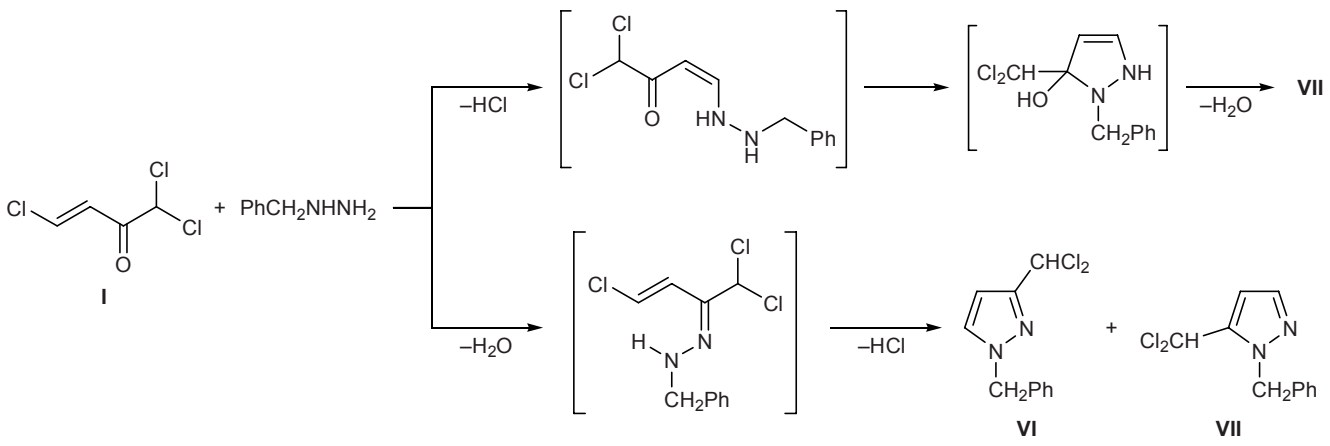
Pyrazoles **VI** and **VII** displayed in the IR spectra narrow peaks due to stretching vibrations of C–H bonds in the pyrazole and benzene rings and alkyl groups and C=C bonds. In the ^{1}H NMR spectra of **VI** and **VII**, the chemical shifts of 4-H were similar, while the 3-H proton in 5-substituted pyrazole **VII** resonated in a stronger field than the 5-H proton in 3-substituted pyrazole **VI**. The mass spectra of 1-benzyl-3(5)-dichloromethyl-1*H*-pyrazoles **VI** and **VII** contained fairly strong molecular ion peaks, and the fragmentation patterns were consistent with published data for pyrazoles [10].

To conclude, we have synthesized 1,1,4-trichlorobut-3-en-2-one by reaction of chloroacetyl chloride with 1,2-dichloroethene in the presence of aluminum chloride and demonstrated its high reactivity toward nitrogen- and sulfur-centered nucleophiles and N,N-binucleophiles.

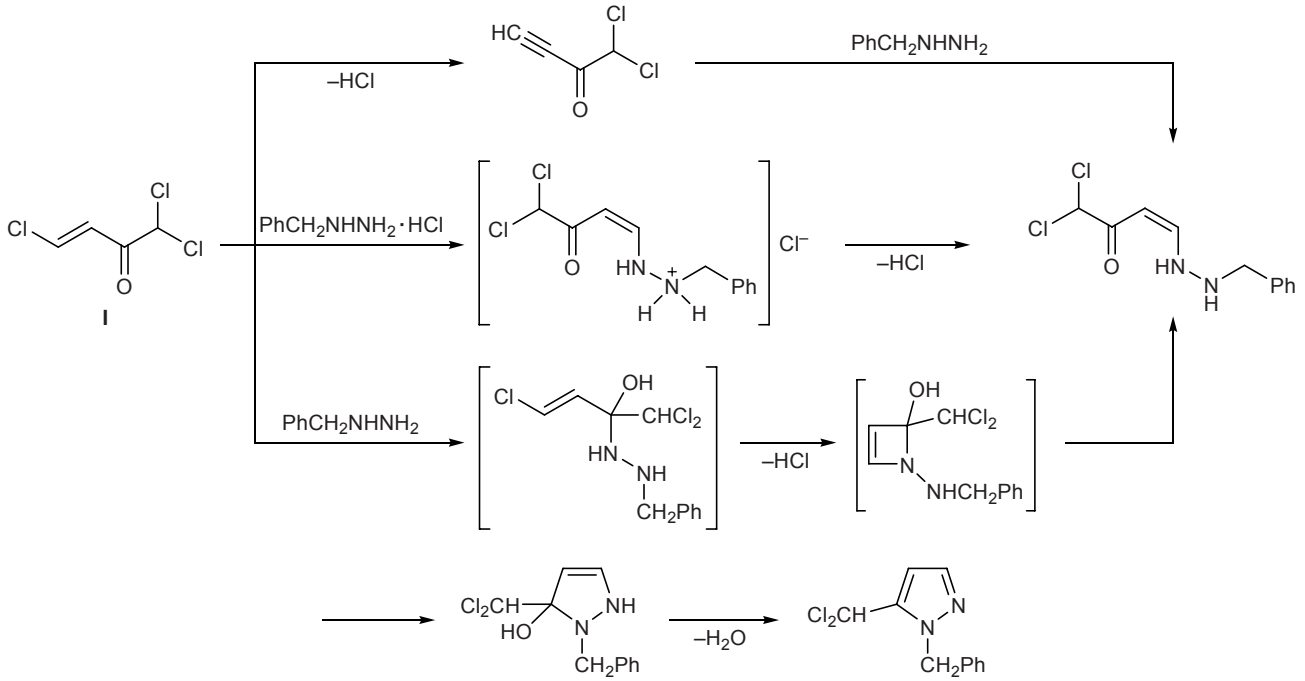
EXPERIMENTAL

The IR spectra were recorded in KBr on a Specord 75IR spectrometer. The ^{1}H and ^{13}C NMR spectra were measured on a Bruker DPX-400 spectrometer at

Scheme 10.



Scheme 11.



400.13 and 100.61 MHz, respectively, using hexamethyldisiloxane as internal reference. The mass spectra were obtained on a Shimadzu GCMS-QP5050A instrument.

1,1,4-Trichlorobut-3-en-2-one (I). *a*. Chloroacetyl chloride, 70.86 g (0.62 mol), was added dropwise under stirring to a solution of 82.67 g (0.62 mol) of aluminum chloride in 100 ml of 1,2-dichloroethylene. The mixture was stirred for 6–8 h at 60°C, cooled, and poured onto ice. The organic layer was separated, and the aqueous phase was extracted with methylene chloride. The extracts were combined with the organic phase and dried over $CaCl_2$, the drying agent was filtered off, the solvent was distilled off, and the residue was distilled under reduced pressure. Yield 75 g (70%), bp 57–60°C (7 mm), n_D^{20} = 1.5262. IR spectrum, ν, cm^{-1}: 1590, 1570 (C=C); 1705, 1690 (C=O); 2990 ($CHCl_2$); 3020, 3080 (=C–H). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 5.88 s (1H, $CHCl_2$), 6.99 d (1H, =CHCO, J = 13.4 Hz), 7.57 d (1H, =CHCl). ^{13}C NMR spectrum, δ_C, ppm: 69.09 ($CHCl_2$), 124.59 (=**C**HCO), 141.82 (=CHCl), 183.12 (C=O). Mass spectrum, *m*/*z* (I_{rel}, %): 173 (5) $[M]^+$, 91 (60), 89 (100), 83 (10), 63 (20), 61 (50), 48 (10). Found, %: C 27.63; H 1.76; Cl 61.30. $C_4H_3Cl_3O$. Calculated, %: C 27.70; H 1.74; Cl 61.33.

b. Acetylene was passed over a period of 2 h through a solution of 14.74 g (0.1 mol) of dichloroacetyl chloride in 300 ml of methylene chloride under vigorous stirring at room temperature. The mixture was cooled to 5°C, 13.33 g (0.1 mol) of aluminum chloride was added in three portions, and the mixture was stirred for 3 h at 5°C with continuous bubbling of acetylene. When the reaction was complete, the mixture was poured onto ice. The organic layer was separated, and the aqueous phase was extracted with methylene chloride. The extracts were combined with the organic phase and dried over $CaCl_2$, the drying agent was filtered off, the solvent was distilled off, and the residue was distilled under reduced pressure. Yield 9.7 g (56%). Samples of **I** obtained as described in *a* and *b* were identical in physicochemical properties.

(4,4-Dichloro-3-oxobut-1-en-1-yl)triethylammonium chloride (II). A solution of 3.47 g (0.02 mol) of ketone **I** in 20 ml of diethyl ether was cooled to –45°C, and 2.02 g (0.02 mol) of triethylamine was added dropwise. The precipitate was filtered off and dried under reduced pressure. Yield 3.02 g (70%), mp 210°C. IR spectrum, ν, cm^{-1}: 1650 (C=C); 1730 (C=O); 2980, 2940 (C–H); 3130, 3080 (=C–H); 3407 (N^+–H). Found, %: C 43.57; H 6.78; Cl 38.64; N 4.98. $C_{10}H_{18}Cl_3NO$. Calculated, %: C 43.74; H 6.61; Cl 38.73; N 5.10.

1,1-Dichloro-4-phenylaminobut-3-en-2-one (III). Aniline, 1.4 g (0.015 mol), was slowly (dropwise) added under stirring to a solution of 2.6 g (0.015 mol)

of ketone **I** in 10 ml of ethanol. The mixture was stirred for 6 h, cooled, poured into water, and adjusted to pH 8 by adding a solution of sodium carbonate. The precipitate was filtered off and dried. Yield 2.89 g (84%), mp 93°C. IR spectrum, ν, cm^{-1}: 1598 (C=C); 1639, 1676 (C=O); 2953 ($CHCl_2$); 3040, 3070 (=C–H); 3245, 3446 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: *E* isomer: 5.81 d (1H, =CHCO, J = 12.3 Hz); 6.86 s (1H, $CHCl_2$); 7.05 m, 7.21 m, and 7.34 m (5H, C_6H_5); 8.21 br.t (1H, =C**H**NH); 10.46 br.d (1H, NH, J = 11.78 Hz); *Z* isomer: 5.56 d (1H, =CHCO, J = 7.7 Hz); 6.74 s (1H, $CHCl_2$); 7.13 m, 7.21 m, and 7.34 m (5H, C_6H_5); 8.01 d.d (1H, =C**H**NH, ^3J = 7.7 Hz); 11.36 d (1H, NH, J = 13.06 Hz). Found, %: C 51.93; H 4.01; Cl 30.45; N 6.09. $C_{10}H_9Cl_2NO$. Calculated, %: C 52.20; H 3.94; Cl 30.82; N 6.09.

1-(4,4-Dichloro-3-oxobut-2-en-1-yl)-3,5-dimethyl-1*H*-pyrazole (IV). Ketone **I**, 3.47 g (0.02 mol), was added under stirring to a solution of 4 g (0.04 mol) of 3,5-dimethyl-1*H*-pyrazole in 20 ml of benzene. The precipitate was filtered off and dissolved in water. A solid separated and was filtered off and dried. Yield 3.15 g (68%), mp 67°C. IR spectrum, ν, cm^{-1}: 1580, 1610 (C=N, C=C); 1690 (C=O); 2996, 2923 (C–H), 3084 (=C–H). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.29 s (3H, CH_3), 2.38 s (3H, CH_3), 5.94 s (1H, 4-H), 6.02 s (1H, $CHCl_2$), 7.13 d (1H, =CHCO, J = 13.3 Hz), 7.99 d (1H, 1-CH). Found, %: C 40.11; H 4.98; Cl 33.69; N 13.34. $C_7H_{10}Cl_2N_2O$. Calculated, %: C 40.21; H 4.82; Cl 33.91; N 13.40.

4,4′-Thiobis(1,1-dichlorobut-3-en-2-one) (V). Ketone **I**, 1.73 g (0.01 mol), was added under stirring to a solution of 2.88 g (0.012 mol) of sodium sulfide nonahydrate in 20 ml of ethanol. The mixture was stirred for 6 h at room temperature and poured into water, and the precipitate was filtered off, washed with water, and dried. Yield 2.77 g (90%), mp 105–107°C. IR spectrum, ν, cm^{-1}: 1630 (C=C); 1680 (C=O); 3000 ($CHCl_2$); 3080, 3020 (=C–H). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.89 s (1H, $CHCl_2$), 6.91 d (1H, =CHCO, J = 15.2 Hz), 8.02 d (1H, =CHS). Found, %: C 31.00; H 2.13; Cl 45.94; S 9.84. $C_8H_6Cl_4O_2S$. Calculated, %: C 31.20; H 1.96; Cl 46.04; S 10.41.

1-Benzyl-3-dichloromethyl-1*H*-pyrazole (VI) and 1-benzyl-5-dichloromethyl-1*H*-pyrazole (VII). Benzylhydrazine, 1.22 g (0.01 mol), was added dropwise to a solution of 1.73 g (0.01 mol) of ketone **I** in 50 ml of diethyl ether at a temperature not exceeding –40°C. The mixture was stirred for 15 min at that temperature, 1.01 g (0.01 mol) of triethylamine was added, and the mixture was stirred for 30 min at –40°C, allowed to warm up to 20°C over a period of 30 min, and treated with water. The organic phase was separated, the aqueous phase was extracted with diethyl ether, the extracts were combined with the organic phase and dried over $CaCl_2$, the drying agent was filtered off, the solvent was distilled off, and the residue was distilled under reduced pressure. Yield 11.1 g (64%). IR spectrum, ν, cm^{-1}: 1524, 1585 (C=C, C=N); 2970, 3000 (C–H); 3080, 3130 (=C–H). Found, %: C 54.80; H 4.22; Cl 29.37; N 11.60. $C_{11}H_{10}Cl_2N_2$. Calculated, %: C 54.79; H 4.18; Cl 29.41; N 11.62.

Compound **VI**. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.88 s (1H, $CHCl_2$), 6.50 d (1H, 4-H, J = 2.3 Hz), 6.53 s (2H, CH_2), 7.14–7.28 m (5H, C_6H_5), 7.31 d (1H, 5-H, J = 2.3 Hz). Mass spectrum, m/z (I_{rel}, %): 240 (20) $[M]^+$, 205 (80), 169 (35), 91 (100), 65 (50), 51 (30), 39 (28).

Compound **VII**. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 5.45 s (1H, $CHCl_2$), 6.50 d (1H, 4-H, J = 2.3 Hz), 6.77 s (2H, CH_2), 7.14–7.28 m (5H, C_6H_5), 7.16 d (1H, 3-H, J = 2.3 Hz). Mass spectrum, m/z (I_{rel}, %): 240 (13) $[M]^+$, 205 (12), 169 (30), 91 (100), 65 (23), 51 (18), 39 (17).

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REFERENCES

1. Kochetkov, N.K., *Usp. Khim*., 1955, vol. 24, p. 32; Pohland, A.E. and Benson, W.R., *Chem. Rev*., 1966, vol. 66, p. 161; Levkovskaya, G.G., Bozhenkov, G.V., and Mirskova, A.N., *Izbrannye metody sinteza i modifikatsii geterotsiklov* (Selected Methods of Synthesis and Modification of Heterocycles), Kartsev, V.G., Ed., Moscow: IBS, 2003, vol. 2, p. 284; Moiseev, I.K., Makarova, N.V., and Zemtsova, M.N., *Izv. Vyssh. Uchebn. Zaved., Ser. Khim. Khim. Tekhnol*., 2007, vol. 59, p. 3.
2. Bozhenkov, G.V., Levkovskaya, G.G., Larina, L.I., Ushakov, P.E., Dolgushin, G.V., and Mirskova, A.N., *Russ. J. Org. Chem*., 2004, vol. 40, p. 1583.
3. Prins, H.J. and Haring, H.G., *Recl. Trav. Chim. Pays–Bas*, 1954, vol. 73, p. 479; *Chem. Abstr*., 1955, vol. 49, p. 12265b.
4. Atavin, A.S., Mirskova, A.N., and Levkovskaya, G.G., *Zh. Org. Khim*., 1973, vol. 9, p. 318.

5. Bozhenkov, G.V., Levkovskaya, G.G., Larina, L.A., Evstaf'eva, I.T., and Mirskova, A.N., *Russ. J. Org. Chem*., 2001, vol. 37, p. 644.
6. Voronina, T.A., Chernyavskii, A.I., and Dobrov, I.V., *Zh. Org. Khim*., 1981, vol. 17, p. 918.
7. Mekhryushev, Yu.Ya. and Poluektov, V.A., *Zh. Fiz. Khim*., 1973, vol. 47, p. 1695.
8. Levkovskaya, G.G., Mirskova, A.N., Atavin, A.S., and Kalikhman, I.D., *Zh. Org. Khim*., 1974, vol. 10, p. 2293.
9. Soulen, R.L., Kundiger, D.G., Searles, S., Jr., and Sanchez, R.A., *J. Org. Chem*., 1967, vol. 32, p. 2661.
10. Polyakova, A.A. and Khmel'nitskii, R.A., *Mass-spektrometriya v organicheskoi khimii* (Mass Spectrometry in Organic Chemistry), Leningrad: Khimiya, 1972, p. 367; Zaikin, V.G., Varlamov, A.V., Mikaya, A.I., and Prostakov, N.S., *Osnovy mass-spektrometrii organicheskikh soedinenii* (Principles of Mass Spectrometry of Organic Compounds), Moscow: MAIK Nauka/Interperiodica, 2001, p. 285.