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Synthesis and Properties of 2-Amino-1-chloroenones

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Abstract—Reactions of alkyl-, allyl-, and arylamines and *o*-aminophenol with 1,2-dichlorovinyl alkyl ketones involve replacement of only one chlorine atom in the β-position with respect to the carbonyl group with formation of 2-alkylamino-, 2-allylamino-, and 2-arylamino-1-chlorovinyl ketones. Diamines of the aromatic and aliphatic series react with 1,2-dichlorovinyl alkyl ketone molecules to give *N*,*N*′-bis(2-acyl-2-chlorovinyl)-substituted diamines.

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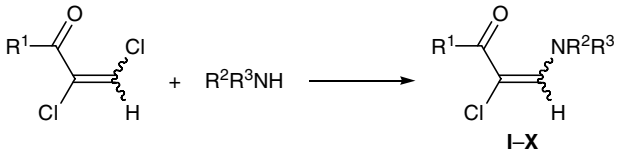
β-Aminovinyl ketones attract attention of many researchers due to their reactivity and sometimes unpredictable behavior. These compounds are used in the synthesis of various heterocyclic systems, dyes, and light-sensitive materials. Different aspects of the chemistry of β-aminovinyl ketones were the subjects of numerous publications, including review articles [1–6]. Methods of synthesis of β-aminovinyl ketones were summarized in [1–3, 5]. In the recent years, procedures are extensively developed for regio- and stereoselective synthesis of aminovinyl ketones containing additional functional groups which could give rise to versatile chemical transformations.

While continuing our studies on the reactivity of alkyl 1,2-dichlorovinyl ketones synthesized previously [7], we examined their reactions with aliphatic and aromatic amines. It is known that reactions of alkylamines with 2-chloro- and 2,2-dichlorovinyl ketones involve replacement of the chlorine atom(s) to give 2-amino- and 2,2-diaminovinyl ketones. Aromatic amines react with alkyl 2,2-dihalovinyl ketones at both halogen atoms and carbonyl group, yielding *N*-aryl-2,2-bis(arylamino)vinyl ketone imines [8–10].

We have found that reactions of alkyl-, allyl-, and arylamines and *o*-aminophenol with alkyl 1,2-dichlorovinyl ketones involve replacement of only one chlorine atom in the β-position with respect to the carbonyl group, leading to 2-alkyl(aryl, allyl)amino-1-chlorovinyl ketones **I**–**V** and **VIII**–**X** (Scheme 1). Diamines of the aromatic and aliphatic series, such as ethane-1,2-diamine and benzene-1,2-diamine, react with two dihalovinyl ketone molecules, and the products are the corresponding *N*,*N*′-bis(2-acyl-2-chlorovinyl) derivatives **VI** and **VII** formed as a result of nucleophilic replacement of the β-chlorine atom in the initial vinyl ketone.

Scheme 1.



I–**X**

I, R^1 = Me, R^2 = H, R^3 = Ph; **II**, R^1 = Me, R^2 = H, R^3 = 4-$MeOC_6H_4$; **III**, R^1 = Me, R^2 = H, R^3 = CH_2=$CHCH_2$; **IV**, R^1 = Pr, R^2 = H, R^3 = Ph; **V**, R^1 = Pr, R^2 = H, R^3 = *t*-Bu; **VI**, R^1 = Pr, R^2 = H, R^3 = $PrC(O)CCl{=}CHNH(CH_2)_2$; **VII**, R^1 = Pr, R^2 = H, R^3 = 4-$[PrC(O)CCl{=}CHNH]C_6H_4$; **VIII**, R^1 = R^2 = R^3 = Pr; **IX**, R^1 = Et, R^2 = Me, R^3 = Ph; **X**, R^1 = Et, R^2 = H, R^3 = 2-HOC_6H_4.

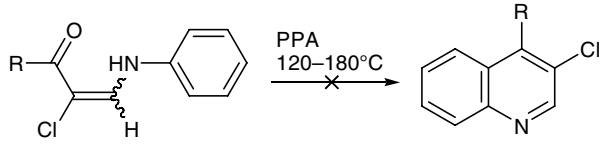
The reactions were carried out in diethyl ether, lower alcohols (methanol, ethanol), and DMSO, the reactant ratio being varied from equimolar to six-fold excess of the amine. The reactions occurred at 0 to 70°C and were accompanied by strong heat evolution. In no case we succeeded in obtaining products of replacement of the chlorine atom in the α-position with respect to the carbonyl group; likewise, no products of amine addition at the carbonyl group of the initial enones were detected.

Compounds **I**–**X** are stable to storage. Despite the presence of N–H and C–Cl bonds, aminovinyl ketones

I–VII and **X** did not undergo cyclization to aziridine derivatives under the examined conditions. The reactions of 1,2-dihalo enones with 1,2-ethylenediamine were not accompanied by intramolecular cyclization of the initially formed 2-amino-1-chlorovinyl ketone to the corresponding 1,2,3,4-tetrahydropyrazine or diazepine. 1-Chloro-2-(2-hydroxyphenyl)aminovinyl ketone **X** failed to undergo cyclization to 3-butyroyl-1,4-benzoxazine (via intramolecular ring closure with participation of the C–Cl and O–H bonds).

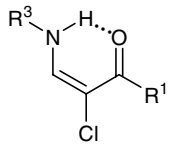
The chemical properties of 2-amino-1-chlorovinyl ketones **I–X** differed from the properties of analogous 2- and 2,2-diaminovinyl ketones having no chlorine atom in the α-position with respect to the carbonyl group [8, 9, 11]. Unlike 2-mono- and 2,2-bis(arylamino)vinyl ketones which are known to undergo intramolecular cyclization to quinoline derivatives on heating in polyphosphoric acid (PPA) [8, 9, 11], heating of compounds **I**, **II**, and **V** in PPA for several hours at 120–180°C did not lead to formation of the corresponding quinolines (Scheme 2).

Scheme 2.

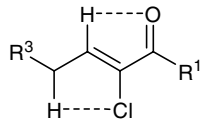


The structure of aminovinyl ketones **I–X** was studied by IR and ^{1}H and ^{13}C NMR spectroscopy and gas chromatography–mass spectrometry. The mass spectra of compounds **V** and **VIII** contained the molecular ion peaks. Compounds **I–X** showed in the IR spectra strong absorption bands belonging to stretching vibrations of the carbonyl groups (1645–1675 cm^{-1}) and C=C (1550–1600 cm^{-1}) and C–H bonds (3020–3090 cm^{-1}); amines **I–VII** and **X** were also characterized by N–H absorption bands at 3250 and 3250–3380 cm^{-1}. The intensity of the C=C band considerably exceeded that of the C=O group; presumably, the reason is strong effect of the fluorine atom and amino group on the electron density distribution in 1-chloro-2-aminoenone molecules.

In the ^{1}H NMR spectra of aminovinyl ketones **I–X** signals from protons in the RC(O) and NR groups and a singlet (**VIII**, **IX**) or doublet (**I–VII**, **X**) from the olefinic protons were present. Analysis of the ^{1}H NMR spectra indicated that compounds **I–X** in solution exist as a single stereoisomer. However, the presence of only one olefinic proton strongly complicated determination of the steric structure of compounds **I–X**. Aminovinyl ketones **I–VII** and **X** containing a secondary amino group can exist as two isomers stabilized by intramolecular hydrogen bonds. 2-Aminovinyl ketones having no substituent in the α-position with respect to the carbonyl group are known to exist as mixtures of *E* and *Z* isomers, while in weakly polar solvents the corresponding *Z* isomers are present exclusively [12–14]. The reason is strong intramolecular hydrogen bonding (six-membered H-chelate ring).



E, *s-cis*



Z, *s-cis*

On the other hand, α-bromo-β-aminovinyl trifluoromethyl ketones were reported to have *Z* (*s-cis*) configuration [6, 15]. The vicinal coupling constant $^3J_{HH}$ between the olefinic proton and proton of the amino group in these compounds is equal to 13–14 Hz. In the ^{1}H NMR spectra of **I–VII** and **X**, the coupling constant $^3J_{HH}$ changes from 12.8 to 13.3 Hz. Taking into account structural similarity between α-chloro-β-aminovinyl ketones **I–X** and α-bromo-β-aminovinyl trifluoromethyl ketones [6], comparison of the corresponding ^{1}H NMR data suggests that aminovinyl ketones **I–VII** and **X** having a secondary amino group in weakly polar solvents ($CDCl_3$) also exist mainly as *Z*/*s-cis*-isomers. It should be noted that the chemical shifts of the NH proton and olefinic proton in the ^{1}H NMR spectra of compounds **I–X** strongly depend on the solvent nature; presumably, this is the result of intermolecular interactions.

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

3-Chloro-4-phenylaminobut-3-en-2-one (I). Aniline, 0.93 g (0.01 mol), was added dropwise under stirring to a solution of 1.39 g (0.01 mol) of 3,4-dichlorobut-3-en-2-one in 15 ml of anhydrous diethyl ether. The mixture was stirred for 2 h, and the precipitate was filtered off, washed with a solution of sodium carbonate and water, and dried. Yield 1.80 g (87%),

mp 135–136°C. IR spectrum, ν, cm^{-1}: 3284 (NH); 3050 (=CH), 3000 (CH_3), 1675 (C=O), 1600 (C=C). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 8.08 d (1H, =CH, J = 13.3 Hz); 7.33 t, 7.08 t, and 7.03 d (5H, H_{arom}, J = 7.7 Hz); 6.95 br.d (1H, NH, J = 13.3 Hz); 3.36 s (3H, CH_3). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 9.16 br.d (1H, NH, J = 12.8); 8.25 d (1H, =CH–, J = 12.8); 7.42 d, 7.32 t, and 7.03 t (5H, H_{arom}, J = 7.7 Hz); 2.34 s (3H, CH_3). ^{13}C NMR spectrum, δ_C, ppm: 192.87 (C=O), 139.52, 135.92, 129.99, 123.94, 116.29, 26.00 (CH_3). Found, %: C 61.33; H 5.30; Cl 18.45; N 7.21. $C_{10}H_{10}ClNO$. Calculated, %: C 61.39; H 5.15; Cl 18.12; N 7.16.

3-Chloro-4-(4-methoxyphenylamino)but-3-en-2-one (II). 3,4-Dichlorobut-3-en-2-one, 1.39 g (0.01 mol), was added dropwise under stirring to a solution of 1.23 g (0.01 mol) of *p*-methoxyaniline in 20 ml of diethyl ether. The mixture was stirred for 1 h on heating under reflux, and the precipitate was filtered off, washed with a solution of sodium carbonate and water, and dried. Yield 1.50 g (77%), mp 113°C. IR spectrum, ν, cm^{-1}: 3250 (NH), 3075 (=CH), 3000 (CH_3), 1675 (C=O), 1600 (C=C). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 7.98 d (1H, =CH, J = 13.3 Hz), 6.99 br.d (1H, NH, J = 13.3 Hz), 6.98 d and 6.87 d (2H each, C_6H_4, J = 8.9 Hz), 3.78 s (3H, OCH_3); 2.34 s (3H, CH_3). Found, %: C 58.51; H 5.46; Cl 15.72; N 6.24. $C_{11}H_{12}ClNO$. Calculated, %: C 58.54; H 5.36; Cl 15.71; N 6.21.

4-Allylamino-3-chlorobut-3-en-2-one (III). 3,4-Dichlorobut-3-en-2-one, 1.39 g (0.01 mol), was added dropwise under stirring to a solution of 1.71 g (0.03 mol) of allylamine in 15 ml of anhydrous diethyl ether. The mixture was stirred for 3 h, the precipitate was filtered off, and the filtrate was washed with a solution of Na_2CO_3 and water, dried over $MgSO_4$, and distilled. Yield 1.25 g (78%), bp 95–100°C (4–5 mm). IR spectrum, ν, cm^{-1}: 3310, 3275 (NH); 3010, 3055, 3090 (=CH); 2910, 2825 (Alk); 1650 (C=O); 1600 (C=C). ^1H NMR spectrum (CCl_4), δ, ppm: *E*/*s*-*cis*: 9.52 br.s (1H, NH), 6.95 d (1H, =CH, J = 12.7 Hz), 6.00 m (1H, =CH), 5.37 t (2H, CH_2), 3.95 t (2H, $=CH_2$, J = 5.5 Hz), 2.32 (3H, CH_3); *Z*/*s*-*cis*: 7.57 d (1H, =CH, J = 12.3 Hz), 6.00 m (1H, =CH), 5.37 t (2H, CH_2), 4.05 t (2H, $=CH_2$, J = 5.7 Hz), 2.31 (3H, CH_3). Found, %: C 52.91; H 6.69; Cl 22.40; N 8.89. $C_7H_{10}ClNO$. Calculated, %: C 52.67; 6.31; Cl 22.21; N 8.78.

2-Chloro-1-phenylaminohex-1-en-3-one (IV). 1,2-Dichlorohex-1-en-3-one, 3.34 g (0.02 mol), was added dropwise under stirring to a solution of 1.87 g (0.02 mol) of aniline in 10 ml of 50% ethanol. The mixture was stirred for 1 h at 60°C, cooled, poured into water, and adjusted to pH 8 by adding a solution of sodium carbonate. The precipitate was filtered off and dried. Yield 3.70 g (85%), mp 74–76°C. IR spectrum, ν, cm^{-1}: 3330 (NH); 3050 (=CH); 2960, 2925, 2865 (C_3H_7); 1635 (C=O); 1610 (C=C). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 8.18 d (1H, =CH, J = 13.2 Hz); 7.39 d, 7.13 t, and 7.09 d (5H, H_{arom}, J = 7.7 Hz); 7.02 br.d (1H, NH, J = 13.2 Hz); 2.72 t (2H, CH_2, J = 7.45 Hz); 1.76 m (2H, CH_2, J = 7.45 Hz); 1.03 t (3H, CH_3, J = 7.45 Hz). ^{13}C NMR spectrum, δ_C, ppm: 192.89 (C=O), 141.65, 139.65, 135.27, 129.95, 123.77, 116.22, 107.25, 40.05 (CH_2), 18.45 (CH_2), 13.96 (CH_3). Found, %: C 64.13; H 6.32; Cl 15.91; N 6.28. $C_{12}H_{14}ClNO$. Calculated, %: C 64.43; H 6.31; Cl 15.85; N 6.26.

1-(*tert*-Butylamino)-2-chlorohex-1-en-3-one (V). 1,2-Dichlorohex-1-en-3-one, 3.34 g (0.02 mol), was added dropwise under stirring to a solution of 4.39 g (0.06 mol) of *tert*-butylamine in 10 ml of 50% ethanol. The mixture was stirred for 2 h at 60°C, cooled, poured into water, adjusted to pH 8 by adding a solution of sodium carbonate, and extracted with diethyl ether. The extract was dried over $CaCl_2$ and evaporated under reduced pressure. Yield 2.44 g (60%). IR spectrum, ν, cm^{-1}: 3370–3275 (NH); 3045 (=CH); 2950, 2920, 2890 (Alk); 1645 (C=O); 1600 (C=C). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 7.73 d (1H, =CH, J = 12.8 Hz), 5.27 br.d (1H, NH, J = 12.8 Hz), 2.53 t (2H, CH_2, J = 7.2 Hz), 1.65 m (2H, CH_2, J = 7.2 Hz), 1.33 s (9H, CH_3), 0.93 t (3H, CH_3, J = 7.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 196.62 (C=O), 146.93, 139.62, 52.48 (**C**Me_3), 30.18 (C**Me**$_3$), 41.22 (CH_2), 18.69 (CH_2), 13.82 (CH_3). Mass spectrum, m/z (I_{rel}, %): 203 (30) $[M]^+$, 188 (20), 160 (8), 146 (10), 132 (10), 121 (20), 119 (75), 104 (80), 71 (12), 57 (100), 43 (32), 42 (40), 41 (95). Found, %: C 59.05; H 8.98; Cl 17.37; N 6.85. $C_{10}H_{18}ClNO$. Calculated, %: C 58.96; H 8.91; Cl 17.40; N 6.88. M 203.71.

***N*,*N*′-Ethylenebis(1-amino-2-chlorohex-1-en-3-one) (VI)** was synthesized from 1.3 g (0.02 mol) of ethane-1,2-diamine in 10 ml of 50% ethanol and 1.67 g (0.01 mol) of 1,2-dichlorohex-1-en-3-one. The mixture was stirred for 2 h at 60°C, cooled, and poured into water, and the precipitate was filtered off and dried. Yield 1.48 g (46%), mp 68–69°C. IR spectrum, ν, cm^{-1}: 3500–3315 (NH); 3045 (=CH); 2970, 2925, 2870 (Alk); 1645 (C=O); 1570 (C=C). ^1H NMR spectrum (acetone-d_6), δ, ppm: 7.78 d (2H, =CH, J = 13.3 Hz), 6.40 br.s (2H, NH), 3.62 t (4H, CH_2, J =

3.3 Hz), 2.44 t (4H, CH_2, J = 7.5 Hz), 1.54 m (4H, CH_2, J = 7.5 Hz), 0.85 t (3H, CH_3, J = 7.5 Hz). ^{13}C NMR spectrum, δ_C, ppm: 189.89 (C=O), 146.84, 49.54 (CH_2); 39.23, 19.48, 14.16 (C_3H_7). Found, %: C 50.14; H 6.96; Cl 21.88; N 8.68. $C_{14}H_{22}Cl_2N_2O_2$. Calculated, %: C 52.34; H 6.90; Cl 22.07; N 8.72.

***N*,*N*′-(*p*-Phenylene)bis(1-amino-2-chlorohex-1-en-3-one) (VII)** was synthesized from 0.22 g (2 mmol) of benzene-1,4-diamine in 15 ml of ethanol and 0.75 g (4 mmol) of 1,2-dichlorohex-1-en-3-one. The mixture was stirred for 1 h at 60°C, cooled, poured into water, and adjusted to pH 8 by adding Na_2CO_3, and the precipitate was filtered off and dried. Yield 0.71 g (96%), mp 194–195°C. IR spectrum, ν, cm^{-1}: 3225 (NH); 3020 (=CH); 2950, 2915, 2855 (C_3H_7); 1650 (C=O); 1545 (C=C). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 9.14 d (2H, NH, J = 12.8 Hz), 8.24 d (2H, =CH, J = 12.8 Hz), 7.40 s (4H, C_6H_4), 2.70 t (4H, CH_2, J = 7.2 Hz), 1.56 m (4H, CH_2, J = 7.2 Hz), 0.90 t (6H, CH_3, J = 7.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 190.21 (C=O), 138.74, 136.22, 117.90, 107.22, 37.90 (CH_2), 18.44 (CH_2), 13.66 (CH_3). Found, %: C 58.56; H 5.84; Cl 19.60; N 8.07. $C_{18}H_{22}Cl_2N_2O_2$. Calculated, %: C 58.54; H 6.00; Cl 19.20; N 7.59.

2-Chloro-1-dipropylaminohex-1-en-3-one (VIII) was synthesized from 3.04 g (0.03 mol) of dipropylamine in 10 ml of 50% ethanol and 1.67 g (0.01 mol) of 1,2-dichlorohex-1-en-3-one. The mixture was stirred for 3 h at 60°C, cooled, poured into water, and extracted with diethyl ether, the extract was dried over $MgSO_4$, the solvent was distilled off, and the residue was kept under reduced pressure. Yield 2.79 g (40%), n_D^{20} = 1.5245. IR spectrum, ν, cm^{-1}: 2950, 2920, 2870 (Alk); 1650 (C=O); 1585, 1565 (C=C). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 7.61 s (1H, =CH), 3.45 t (4H, CH_2, J = 7.5 Hz), 2.52 t (2H, CH_2, J = 7.2 Hz), 1.65 m and 1.55 m (6H, CH_2), 0.88 t and 0.87 t (9H, CH_3). ^{13}C NMR spectrum, δ_C, ppm: 194.35 (C=O), 143.65 (=CH), 39.87 (CH_2), 22.72 (CH_2), 18.51 (CH_2), 13.82 (CH_3), 10.67 (CH_3). Mass spectrum, m/z (I_{rel}, %): 231 (70) $[M]^+$, 202 (23), 188 (18), 160 (12), 72 (12), 71 (48), 70 (30), 43 (100), 41 (51). Found, %: C 62.43; H 9.31; Cl 15.85; N 6.26. $C_{12}H_{22}ClNO$. Calculated, %: C 62.19; H 9.57; Cl 15.30; N 6.04. M 231.77.

2-Chloro-(*N*-methylphenylamino)pent-1-en-3-one (IX). *N*-Methylaniline, 1.07 g (0.01 mol), was added dropwise under stirring to a solution of 1.53 g (0.01 mol) of 1,2-dichloropent-1-en-3-one in 15 ml of DMSO. The mixture was stirred for 2 h, poured into water, adjusted to pH 8 by adding a solution of sodium carbonate, and extracted with methylene chloride. The extract was dried over $CaCl_2$ and evaporated, and the residue was distilled under reduced pressure. Yield 1.56 g (70%), bp 148°C (2 mm). IR spectrum, ν, cm^{-1}: 3050 (=CH); 2930, 2960 (Alk); 1670 (C=O); 1600, 1550 (C=C). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 7.84 s (1H, =CH), 7.34 t and 7.13 m (5H, C_6H_5), 3.65 s (3H, CH_3), 2.68 q (2H, CH_2, J = 7.3 Hz), 1.11 t (3H, CH_3, J = 7.3 Hz). ^{13}C NMR spectrum, δ_C, ppm: 195.99 (C=O), 147.00, 141.56, 129.36, 125.20, 121.37, 116.24, 105.90, 39.76 (CH_2), 31.68 (NCH_3), 9.02 (CH_3). Found, %: C 64.50; H 6.50; Cl 15.90; N 6.25. $C_{12}H_{14}ClNO$. Calculated, %: C 64.43; H 6.31; Cl 15.85; N 6.26.

2-Chloro-1-(2-hydroxyphenylamino)pent-1-en-3-one (X) was synthesized from 1.09 g (0.01 mol) of 2-aminophenol in 15 ml of ethanol and 1.53 g (0.01 mol) of 1,2-dichloropent-1-en-3-one. The mixture was stirred for 2 h at 40°C, cooled, poured into water, and adjusted to pH 7–8, and the precipitate was filtered off and dried. Yield 1.75 g (68%), mp 161–162°C. IR spectrum, ν, cm^{-1}: 3355 (NH); 3200–3300 (OH); 2965, 2925 (C_2H_5); 1650 (C=O); 1600, 1550 (C=C). ^1H NMR spectrum (acetone-d_6), δ, ppm: 9.17 s (1H, OH), 8.39 d (1H, =CH, J = 13.3 Hz), 7.59 br.d (1H, NH, J = 13.3 Hz), 7.38 m and 6.88 m (4H, C_6H_4), 2.76 q (2H, CH_2, J = 7.3 Hz), 1.08 t (3H, CH_3, J = 7.3 Hz). Found, %: C 59.45; H 5.62; Cl 15.72; N 6.76. $C_{11}H_{12}ClNO$. Calculated, %: C 58.54; H 5.36; Cl 15.71; N 6.21.

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