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REACTIONS OF CYCLOHEXYL-SUBSTITUTED UNSATURATED MONO- AND SATURATED DICHLOROKETONES WITH ETHYLENEDIAMINE

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ABSTRACT

Electrophilic addition of cyclohexane- and chloro-substituted cyclohexanecarboxylic acid chlorides to 3-chloro-1-propene and 2-methyl-3-chloro-1-propenes in the presence of an $AlCl_3$ *catalyst at –15 ÷ –20°C yielded 1-cyclohexyl(chlorocyclohexyl)-3-H(*CH_3*)-3,4-dichloro-1-butanones. These were then dehydrochlorinated in a superbasic medium (DMFA +* Na_2CO_3*) to form 1-cyclohexyl (chlorocyclohexyl)-3-H(*CH_3*)-4-chloro-2-buten-1-ones. Mono- and dipyrrole compounds were synthesized by reacting cycloalkyl-substituted saturated dichloroketones and unsaturated chloroketones with ethylenediamine in an aqueous alkaline medium, depending on the ratio of the starting materials. The structures of the resulting mono- and dipyrroles were confirmed by IR and* ^1H *NMR spectroscopy.*

INTRODUCTION

A pyrrole fragment is found in the composition of many natural and synthetic biologically active compounds, catalysts, drugs, etc. compounds [1–9]. Examples of these include vitamin B_{12}, bile pigments bilirubin and biliverdin, blood pigments heme, chlorophyll, etc. [3, 4]. Alkoxy derivatives of pyrroles have a wide range of applications as optically active compounds [10, 11]. Polymers such as polypyrrole exhibit unique electrophysical and optical properties [1, 12]. Polypyrroles are also widely used in the preparation of conventional and solar cells [13].

The wide range of applications of pyrroles has given a strong impetus to research on their synthesis by new methods.

Saturated and unsaturated chloroketones belong to a class of promising starting compounds for the synthesis of heterocyclic compounds with one or two heteroatoms [14-24]. Electrophilic addition reactions of carboxylic acid chloranhydrides to unsaturated hydrocarbons, their chlorine-substituted derivatives, and allyl chlorides are among the most favorable reactions for the synthesis of saturated and unsaturated chloroketones [15, 17–19, 21, 23–27].

It is known that pyrrole compounds are formed when the products of electrophilic addition of alkanecarboxylic acid chlorhydrides to allyl halides are reacted with monoamines [28–30]. The advantage of this new synthesis method, called the Kost-Ibrahimov-Mammadov method [31], is that, depending on the structure of the starting mono-amines, various functional groups can be introduced into the pyrrole ring, as well as one or more pyrrole rings can be formed in the molecule according to the basicity of the carbon atom taken [27–30, 32, 33].

The aim of the presented work is to synthesize compounds containing one or two pyrrole rings by reacting the products of electrophilic addition of monobasic cycloalkanocarboxylic acid chloranhydrides (CACAChA) to 3-chloropropene and 2-methyl-3-chloropropene with ethylenediamine (EDA).

EXPERIMENTAL

IR spectra were recorded on a Thermo Scientific Nicolet IS10FT-IR spectrophotometer, and NMR ^{1}H spectra were recorded on a Bruker AM-360 Spectrometer (internal standard TMS or HMDC).

Commercially available reagents such as 3-chloro-1-propene, 2-methyl-3-chloro-1-propene, and EDA were used as starting materials, and CACAChA were obtained by the reaction of the corresponding acids with PCl_3 or $SOCl_2$.

1. Synthesis of 1,2-di[2-cycloalkyl-4-H(CH$_3$)pyrrolyl-1]ethanes (5a-c, 6a-c). To a solution of 0.05 mol (3g) EDA and 0.2 mol (8g) NaOH in water, 0.1 mol of dichloroketones 1a-c (2a-c) is added at –5°C. The reaction mixture is heated at +60 ÷ +70°C for 4 hours, cooled and extracted with ether (3x100ml). The ether extract is washed with water, 10% Na_2CO_3 and again with water and dried over anhydrous Na_2SO_4. The solvent is removed with a water pump, and the residue is distilled under vacuum in a nitrogen atmosphere to obtain dipyrroles 5a-c (6a-c) (scheme 2). The obtained dipyrroles crystallize upon storage and are recrystallized from methanol or ethanol.

1.1. 1,2-Di(2-cyclohexylpyrrolyl-1]ethane (5a). T_m (melting temperature): 90–92^{0}C (C_2H_5OH), yield 58%. IR spectrum (cm^{-1}): 3114 (ν=CH, pyrrole), 1514, 1601 (*ν*C=C,C=C pyrrole). NMR ^{1}H spectrum (δ, m.h.): 1.0 – 2.1 m (22 H, cyclohexane), 4.07 s (4H, NCH_2-CH_2), 5.81-5.86 m (2H, C^3–H pyrrole), 6.02–6.07 m (2H, C^4–H pyrrole), 6.34–6.37 m (2H, C^5-H pyrrole). Found., %: C 81.56, H 9.80 8.59. $C_{22}H_{32}N_2$. Calc.,%: C 81.48, H 9.88, N 8.64.

1.2. 1,2-Di[2-(1-chlorocyclohexyl)pyrrolyl-1]ethane (5b). T_m: 101–103^{0}C (C_2H_5OH), yield 52%. IR spectrum (cm^{-1}): 3030 (ν=CH, pyrrole), 1504, 1605 (*ν*C=C,C=C pyrrole). Found., %: C 67.09, H 7.58, Cl 18.19, N 7.06. $C_{22}H_{30}Cl_2N_2$. Calc., %: C 67.18, H 7.63, Cl 18.07, N 7.12.

1.3. 1,2-Di[2-(4-chlorocyclohexyl) pyrrolyl-1]ethane (5c). T_m: 108–109^{0}C (C_2H_5OH), yield 54%. IR spectrum (cm^{-1}): 3025 (*ν*=CH, pyrrole), 1500, 1575 (*ν*C=C,C=C pyrrole). Found., %: C 67.26, H 7.68, Cl 17.98, N 7.08. $C_{22}H_{30}Cl_2N_2$. Calc., %: C 67.18, H 7.63, Cl 18.07, N 7.12.

1.4. 1,2-Di(2-cyclohexyl-4-methylpyrrolyl-1)ethane (6a). T_m: 152–154^{0}C (C_2H_5OH), yield 61%. IR spectrum (cm^{-1}): 3092 (*ν*=CH, pyrrole), 1505, 1585 (*ν*C=C,C=C pyrrole). NMR ^{1}H spectrum (δ, m.h.): 0.8–2.0 m (22 H, cyclohexane), 1.86 s (6H, 2CH_3), 4.0 s (4H, NCH_2-CH_2), 5.76–5.81 m (2H, C^3-H pyrrole), 6.41–6.52 m (2H, C^5–H pyrrole). Found., %: C 81.71, H 10.16, N 8.08. $C_{24}H_{36}N_2$. Calc., %: C 81.82, H 10.23, N 7.95.

1.5. 1,2-Di[2-(1-chlorocyclohexyl)-4-methylpyrrolyl-1]ethane (6b). T_m: 142–144^{0}C (CH_3OH), yield 54%. IR spectrum (cm-1): 3120 (*ν*=CH, pyrrole), 1516, 1600 (*ν*C=C,C=C pyrrole). Found, %:

C 68.32, H 8.17, Cl 16.98. N 6.53. $C_{24}H_{34}Cl_2N_2$. Calc., %: C 68.41, H 8.08, Cl 16.86, N 6.65.

1.6. 1,2-Di[2-(4-chlorocyclohexyl)-4-methylpyrrolyl-1]ethane (6c). T_m: 146–148^{0}C (CH_3OH), yield 56%. IR spectrum (cm-1): 3020 (ν=CH, pyrrole), 1510, 1596 (νC=C,C=C pyrrole). Found, %: C 68.32, H 8.17, Cl 16.98. N 6.53. $C_{24}H_{34}Cl_2N_2$. Calc., %: C 68.41, H 8.08, Cl 16.86, N 6.65.

2. Preparation of 1-(2-aminoethyl)-2-cycloalkyl-4-H(CH_3)pyrroles (7a-c, 8a-c). Monopyrroles 7a-c (8a-c) were obtained by the reaction of 0.05 mol (3g) EDA and 0.1 mol (4g) NaOH in water with 0.1 mol of saturated dichloroketones 1a-c (2a-c) using method 1 above (scheme 2).

2.1. 1-(2-aminoethyl)-2-cyclohexylpyrrole (7a). T_b (boiling temperature): 125–126^{0}C (5), n_D^{20} 1.5295, d_4^{20} 1.0181, yield 58%. IR spectrum (cm^{-1}): 3310, 3446 (νNH_2), 3112 (ν=CH, pyrrole), 1514, 1592 (νC=C,C=C pyrrole). NMR ^1H spectrum (δ, m.h.): 1.0-2.2 m (11H, cyclohexane), 1.75 s (2H, NH_2), 2.55 t (2H, CH_2N, J=7), 3.42 t (2H, NCH_2 J=7), 5.51–5.59 m (1H, C^3-H pyrrole), 5.7-5.9 m (1H, C^4–H pyrrole), 6.22–6.31 m (1H, C^5–H pyrrole). Found., %: C 75.08, H 10.37, N 14.52. $C_{12}H_{20}N_2$. Calc., %: C 75.00, H 10.42, N 14.58.

2.2. 1-(2-aminoethyl)-2-(1-chlorocyclohexyl)pyrrole (7b). T_b: 148–150^{0}C (3), n_D^{20} 1.5430, d_4^{20} 1.1162, yield 49%. IR spectrum (cm^{-1}): 3350, 3450 (νNH_2), 3140 (ν=CH, pyrrole), 1510, 1605 (νC=C,C=C pyrrole). Found, %: C 63.42, H 8.27, Cl 15.81. N 12.24. $C_{12}H_{19}ClN_2$. Calc., %: C 63.58, H 8.39, Cl 15.67. N 12.36.

2.3. 1-(2-aminoethyl)-2-(4-chlorocyclohexyl)pyrrole (7c). T_b: 146–147^{0}C (4), n_D^{20} 1.5425, d_4^{20} 1.1158, yield 51%. IR spectrum (cm^{-1}): 3330, 3442 (νNH_2), 3114 (ν=CH, pyrrole), 1505, 1597 (νC=C,C=C pyrrole). Found., %: C 63.71, H 8.46, Cl 15.43. N 12.41. $C_{12}H_{19}ClN_2$. Calc., %: C 63.58, H 8.39, Cl 15.67. N 12.36.

2.4. 1-(2-aminoethyl)-2-cyclohexyl-4-methylpyrrole (8a). T_m: 141–143^{0}C (3), n_D^{20} 1.5260, d_4^{20} 0.9936, yield 56%. IR spectrum (cm^{-1}): 3305, 3440 (νNH2), 3045 (ν=CH, pyrrole), 1500, 1568 (νC=C,C=C pyrrole). NMR ^1H spectrum (δ, m.h.): 1.1-2.3 m (11H, cyclohexane), 1.74 s (2H, NH_2), 1.94 s (3H, CH_3), 2.54 t (2H, CH_2N, J=7), 3.44 t (2H, NCH_2, J=7), 5.60–5.65 m (1H, C^3-H pyrrole), 6.34–6.41 m (1H, C^5-H). Found., %: C 75.82, H 10.59, N 13.47. $C_{13}H_{22}N_2$. Calc., %: C 75.73, H 10.68, N 13.59.

2.5. 1-(2-aminoethyl)-2-(1-chlorocyclohexyl)-4-methylpyrrole (8b). T_b: 154–156^{0}C (2), n_D^{20} 1.5422, d_4^{20} 1.1141. IR spectrum (cm^{-1}): 3340, 3448 (νNH_2), 3120 (ν=CH, pyrrole), 1520, 1601 (νC=C,C=C pyrrole). Found., %: C 64.73, H 8.62, Cl 14.91, N 11.54. $C_{12}H_{19}ClN_2$. Calc., %: C 64.87, H 8.73, Cl 14.76, N 11.64.

2.6. 1-(2-aminoethyl)-2-(4-chlorocyclohexyl)-4-methylpyrrole (8c). T_b: 158–160^{0}C (4), n_D^{20} 1.5410, d_4^{20} 1.1126. IR spectrum (cm^{-1}): 3326, 3440 (νNH_2), 3095 (ν=CH, pyrrole), 1505, 1594 (νC=C,C=C pyrrole). Found., %: C 64.98, H 8.86, Cl 14.58, N 11.51. $C_{12}H_{19}ClN_2$. Calc., %: C 64.87, H 8.73, Cl 14.76, N 11.64.

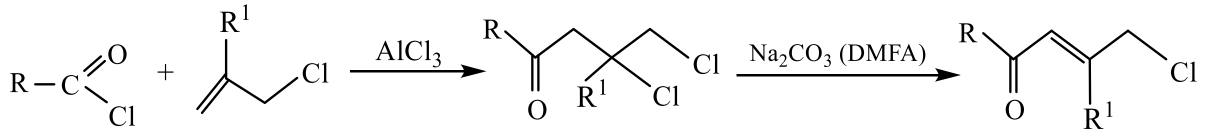
RESULTS AND DISCUSSION

Compounds containing one or two heteroatom-containing five-membered heterocyclic rings are widely found in nature [34, 35], therefore, the synthesis of these compounds by new methods and the study of their structures are one of the most important theoretical and applied problems of modern organic chemistry.

For the synthesis of cycloalkyl-substituted mono- and dipyrroles, the reactions of the electrophilic addition products of CACAChA to 3-chloro-1-propene and 2-methyl-3-chloro-1-propene with EDA were studied.

As a result of electrophilic coupling reactions, the corresponding 1-cycloalkyl-3-H(CH_3)-3,4-dichloro-1-butanones (1a-c, 2a-c) were obtained, and their dehydrochlorination in a super alkaline medium (DMFA+Na_2CO_3) at a temperature of +80 ÷ +85% gave 1-cycloalkyl-3-H(CH_3)-4-chloro-2-buten-1-ones (3a-c, 4a-c) (scheme 1) [15, 17, 23, 27].

Scheme 1.



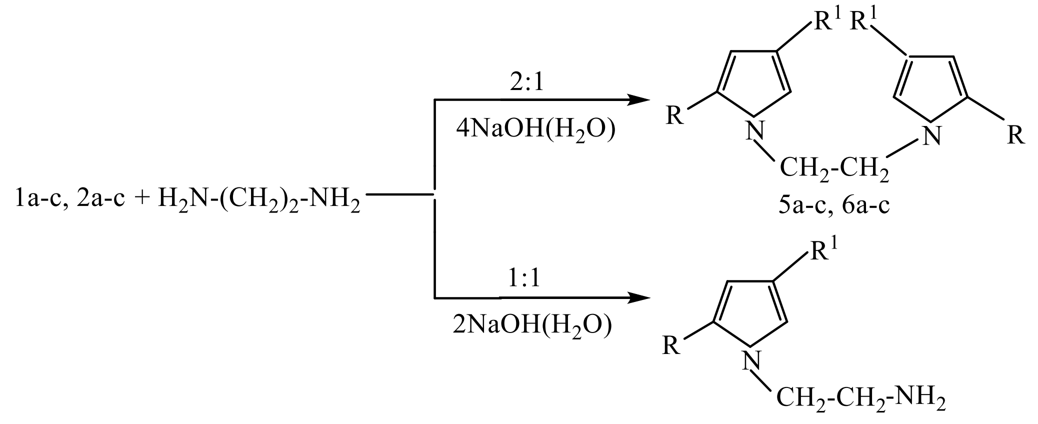
1a-c, 2a-c

3a-c, 4a-c

Here, R = *ts*-C_6H_{11} (a), 1-Cl-*ts*-C_6H_{10} (b), 4-Cl-*ts*-C_6H_{10} (c); R^1 = H (1a-c, 3a-c), CH_3 (2a-c, 4a-c).

During the reaction of cycloalkyl-substituted saturated dichloroketones (1a-c, 2a-c) and unsaturated chloroketones (3a-c, 4a-c) with EDA in an aqueous-alkaline medium, it was determined that, for example, dichloroketones 1a-c, 2a-c, in an aqueous solution of starting materials in a 2:1 ratio (1a-c or 2a-c : EDA - 2:1) and 4 equivalents of NaOH, mainly 1,2-di[2-cycloalkyl-4-H(CH_3)pyrrolyl-1]ethane dipyrroles (5a-c, 6a-c) are obtained, and in an aqueous solution of 1:1 ratio (1a-c or 2a-c : EDA - 1:1) and 2 equivalents of NaOH, 1-(2-aminoethyl)-2-cycloalkyl-4-H(CH_3)pyrroles (7a-c, 8a-c) are obtained (scheme 2).

Scheme 2.



5a-c, 6a-c

7a-c, 8a-c

Here, R - as shown in scheme 1; R^1 = H (5a-c,7a-c), CH_3 (6a-c, 8a-c).

Unsaturated chloroketones 3a-c, 4a-c also react with EDA in aqueous medium in a 2:1 ratio of starting materials (3a-c or 4a-c : EDA - 2:1) and in the presence of 2 equivalents of NaOH, dipyrrole compounds 5a-c, 6a-c are formed, and monopyrroles 7a-c, 8a-c are formed in equimolar amounts of all reagents.

The structure of the synthesized dipyrroles and monopyrroles was confirmed by IR and NMR ^1H spectra.

In the IR spectra of pyrroles 5a-c – 8a-c, characteristic absorption bands for the pyrrole ring were determined (cm^{-1}): 3030–3140(ν=CH), 1500–1520 (νC=C), 1550–1605 (νC=C). In addition, in monopyrroles 7a-c, 8a-c, the absorption band of the valence vibration of the amine group appears in the regions 3305–3350 and 3440–3450 cm^{-1}.

In the NMR ^{1}H spectra of dipyrroles (5a-c, 6a-c) and monopyrroles (7a-c, 8a-c), the protons of the pyrrole ring appear in the form of a characteristic multiplet (m) in the regions 5.50–6.52 m.h. Also, in dipyrroles 5a-c, 6a-c the protons of the substituted ethane fragment are observed as singlets (s) in the 4.0-4.07 m.h. regions, and in monopyrroles 7a-c, 8a-c these protons are observed as two triplets (t) in the 2.54–2.55 and 3.42–3.44 m.h. absorption regions.

It should be noted that the dichloroketones 1a-c, 2a-c used in the synthesis of pyrroles do not require special separation from the reaction products and additional purification, they were used as crude products. Since they are easily heterocyclized to furan compounds during distillation under vacuum [15, 36].

CONCLUSION

Cycloalkyl-substituted saturated dichloroketones 1a-c, 2a-c were obtained from electrophilic coupling reactions of CACAChA to 3-chloro-1-propene and 2-methyl-3-chloro-1-propene in the presence of $AlCl_3$ catalyst at a temperature of -15 ÷ -20°C in dichloroethane medium, and unsaturated chloroketones 3a-c, 4a-c were synthesized from their dehydrochlorination in a super-alkaline medium (DMFA+Na_2CO_3) (scheme 1). During the reaction of saturated dichloroketones 1a-c, 2a-c and unsaturated chloroketones 3a-c, 4a-c with EDA in an aqueous-alkaline (H_2O+NaOH) medium, depending on the ratio of the starting materials, 5a-c, 6a-c dipyrroles and 7a-c, 8a-c monopyrroles (scheme 2) were obtained.

The obtained experimental results contributed to the introduction of new information into the theoretical issues of organic chemistry. It was established that in the DMFA+Na_2CO_3 super alkaline medium (scheme 1), under the influence of DEA and an aqueous solution of alkali (H_2O+NaOH) (scheme 2), the chlorine atoms in the cyclohexyl ring do not undergo any chemical transformations, that is, they do not enter into nucleophilic (S_N) or dehydrochlorination reactions.

Thus, a simple and convenient method for the preparation of monopyrrole and dipyrrole compounds from the interaction of the electrophilic addition products of CACAChA to 3-chloro-1-propene and 2-methyl-3-chloro-1-propene with EDA has been proposed.

Conflicts of Interest

The authors declare no conflict of interest.

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