

Chloracetylation Reaction of Alkylanilines

Abdushukurov Anvar Kabirovich

Professor of the Faculty of Chemistry of the National University of Uzbekistan

Saidova Xurshida Azimovna

Graduate of the Faculty of chemistry of the National University of Uzbekistan

Islamova Yulduz O'ralovna

Teacher of the Faculty of chemistry of the National University of Uzbekistan

Mamatkulov Nematillo Narzullaevich

Associate Professor of the Faculty of Chemistry of the National University of Uzbekistan

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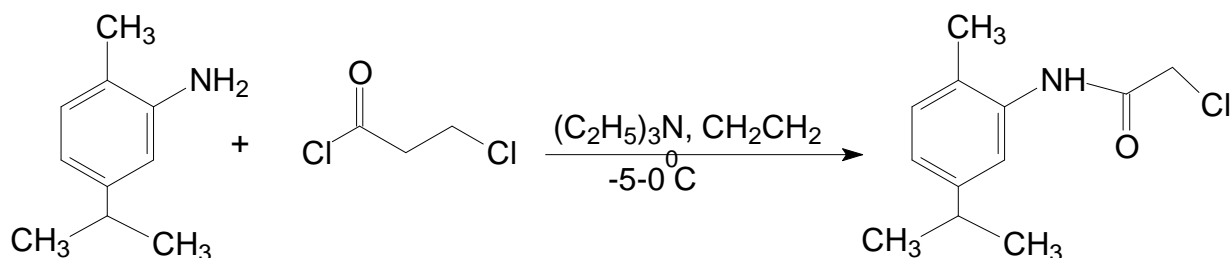
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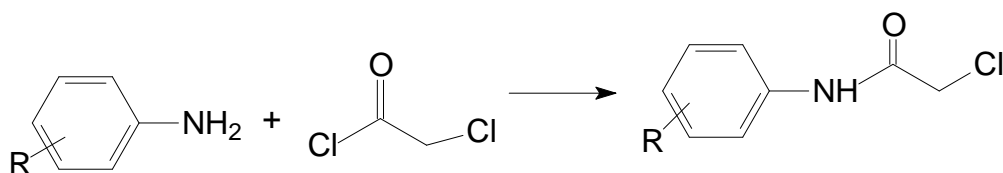
ABSTRACT

This article studied the reaction of ethylaniline and dimethylaniline with chloroacetyl chloride in a solution of benzene and acetone. New organic substances that are not known in the literature have been synthesized. The structure of substances has been proven through the yamr Spectra on IQ, H1, and YaMR on C¹³.

In literature data, biologically active substances and melanogenesis inhibitors were synthesized by the reaction of 2-Methyl-5-isopropylaniline with chloroacetyl chloride [1-2].

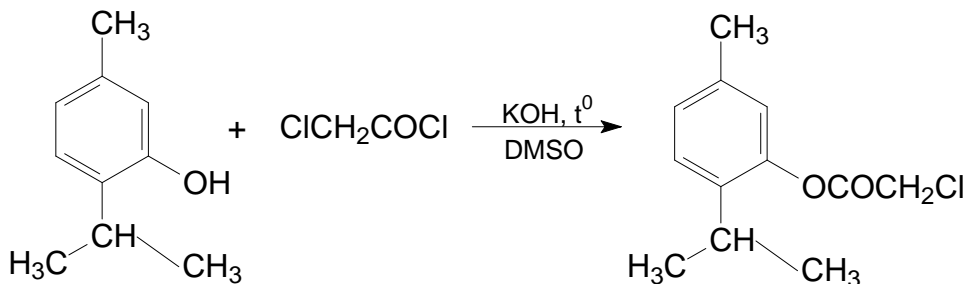


Compounds with biological phaolicity have been obtained by scientists as a result of the reaction of chloroacetyl chloride with 3-, 4- exchangeable aminobiricles [3-4].

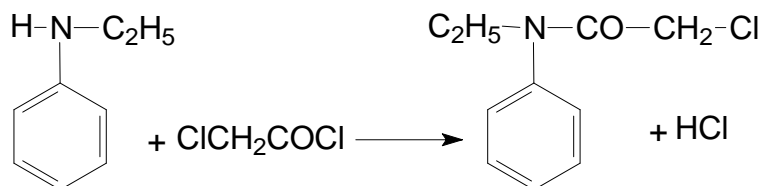


R=H, 3-Cl, 4-Cl, 2-NO₂, 4-CH₃, 4-PCH₃, 4-CF₃, 3-CF, 2-COOH, 4-COOH, 2-F, 4-F, 4-Br.

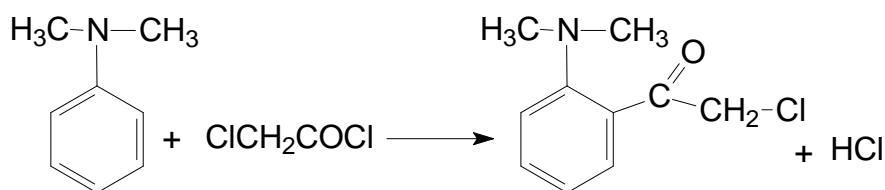
The authors conducted chloroacetylation reactions with 5-methyl-2-(1-methylethyl)phenol under different conditions for the purpose of synthesizing biological faolic substances. The highest unum, 5-methyl-2-(1-methylethyl)phenylchloracetet, was found. The reaction goes according to the following scheme [5-6].



No chlorine acetylation reactions of N-alkylaniline derivatives have been studied in literature data. For this reason, reactions of ethylaniline and dimethylanilines with chloroacetyl chloride in benzene, acetone solution were carried out to synthesize new organic compounds. The reaction proceeds with ethylaniline according to the following scheme to form N-ethyl - N-chloroacetylaniline.



Chloroacetylation reactions with dimethylaniline have also been carried out under similar conditions. The reaction revealed that the product was N-, N-dimethyl(amino) phenacyl chloride.



Experimental part

N-ethyl-N-chloroacetyl chloride centesis

Experience № 1. N-ethyl-N-chloroacetyl chloride centesis. A 50 ml tube with a reverse cooler, thermometer, mixer and dropper funnel was placed in a circular tube ice bath. 20 ml of 6.05 gr (0.05 mol) n-ethylaniline and benzene solvent were placed in the flask. Then a drop of 4 ml of chloroacetyl chloride was slowly dripped into the flask through a funnel for 45 minutes. In this case, in a device equipped with a mixer, the reaction process was carried out with cooling during mixing. The reaction was carried out for 5 hours. The reaction was found to be over by stopping the release of HCl. In the reaction process passed with the formation of a white precipitate. The sediment was washed away in acetone. N-ethyl-N-chloroacetyl chloride Unum 5.6 gr (58%)

In the IR spectrum of N-ethyl-N-chloroacetyl chloride, we can observe the absorption lines of the AR-H bond belonging to the aromatic ring in the 30-63 cm^{-1} field. In addition, we can see that the absorption lines of the C-H bond linked to the sp^3 hybridized C atom in the 2969-2882 cm^{-1} field range in the chain and in the 2805-2711 cm^{-1} range and the N-C-H bond in the 1438 cm^{-1} field demonstrated intensive signals. In the range of fields 1602 cm^{-1} -1583 cm^{-1} , we can observe the suction lines belonging to the third amid garden. Absorption lines produced by Valence fluctuations of C=O 17-12 cm^{-1} binding has been observed in the domain. Absorption lines of the C-Cl bond bound to the sp^3 hybridized C atom in the chain have been observed in the 750-688 cm^{-1} field range.

In the ^1H NMR analysis of n-ethyl-n-chloroacetyl chloride on H^1 , triplet signals can be seen for 1.542 ppm, 1.549 ppm and 1.557 ppm for the R- CH_3 bond of carbon-bonded hydrogen sp^3 hybridized methyl group. quartet signaling has been observed at 3.451373 ppm, 3.451380 ppm, 3.451388 ppm, 3.451388 ppm, and 3.451395 ppm. for the N- CH_2 -R bond of hydrogen, the methylene group is hybridized here sp^3 and bound to the nitrogen atom. we can observe that the Cl-C-H bond of the carbon in the methylene group that formed the bond with the sp^3 hybridized chlorine atom recorded a singlet signal at 3.31324 ppm. The signals of the carbon-bonded AR-H hydrogen in the aromatic ring are Doublet at 7,590 ppm, triplet signals at 7,576 ppm, and triplet signals at 7,559 ppm.

In a ^{13}C NMR analysis of N-ethyl-n-chloroacetyl chloride on C^{13} , the signal of the R- CH_3 bond of the sp^3 hybridized carbon was observed at 11.367 ppm, as well as a signal at N- CH_2 -R carbon 48.714 ppm in the methylene group bound to nitrogen. The Ar-C bond in the aromatic ring can observe multiplet signals at 123, 131, 130, 136 PPMS. we can see that a signal was observed at 49.0 ppm at the expense of the carbon Cl-C-H bond in the methylene group that formed a bond with the sp^3 hybridized chlorine atom. The carbon of the N-C=O bond in the carbonyl group bound to the nitrogen atom was to be observed in the 155-185 ppm range, with no such field given in the spectrum.

Experience № 2. Synthesis of N -, N-dimethyl (amino) phenacyl chloride. A 50 ml tube with a reverse cooler, thermometer, mixer and dropper funnel was placed in a round tube ice bath. The flask was infused with 6.05 ml (0.05 mol) of dimethylaniline and 20 ml of acetone solvent. Then 4 ml of chloroacetylchloride droplet was added to the flask slowly drip through the funnel for 40 minutes. In this case, in a device equipped with a mixer, the reaction process was carried out with cooling while stirring. The reaction took 6 hours. It was determined that the reaction was over by the cessation of HCl release. From the reactionary mixture, acetone was expelled under normal conditions. And the substance was expelled in vacuum at 230-2350 $^{\circ}\text{C}$ / 20 mm.sim.ust. N -, N-dimethyl(amino) phenacyl chloride Unum 4.8 gr (52%).

The absorption lines of the AR-H bond bound to the sp^2 hybridized C atom in the N -, n-dimethyl(amino) phenacyl chloride IR spectrum 3055-3024 cm^{-1} aromatic ring, absorption lines of the C=O bond formed from Valence fluctuations in the 1748 cm^{-1} field, and absorption lines of the C-H bond bound to the sp^3 hybridized C atom are in the 2930-2879 cm^{-1} field range. In the 692 cm^{-1} field, the absorption line of the C-Cl bond is observed, which is bound by the sp^3 hybridized C atom. We can also see the suction lines generated by the N- CH_3 garden deformation fluctuations in the areas of 1495-1468 cm^{-1} . The n, n-dimethyl group directs the aromatic ring to the para state, and in the 866 cm^{-1} field we can observe the absorption lines of the exactly 1.4 substituted state of the benzene ring.

A ^1H NMR analysis of N -, N-dimethyl(amino) phenacyl chloride on H^1 observed a singlet signal at 2.905 ppm for the N- CH_3 bond of hydrogen in the methyl group where sp^3 is hybridized and bound to a nitrogen atom. we can observe that hydrogen bonded to carbon in the methylene Cl-C-H group, which sp^3 hybridized and formed a bond with the chlorine atom, recorded a singlet signal at 3.136 ppm. The signals of Ar-H hydrogen bound to carbon in the aromatic ring

are triplet at 7.492 ppm and 7.377 ppm, double signals at 7.610 ppm and 7.808 ppm. Based on the signals in the benzene ring, we can conclude that the reactionary product is oriented towards the ortho state rather than the para state with respect to the N, N-dimethyl group.

In a ¹H NMR analysis of N, N-dimethyl(amino) phenacyl chloride observed a singlet signal for H¹ on the 2.905 ppm n-CH₃ bond hydrogen in the methyl group where sp³ is hybridized and bound to a nitrogen atom. we can observe that the carbon-bonded Hydrogen of the methylene Cl-C-H group, which sp³ hybridized and formed a bond with the chlorine atom, recorded a singlet signal at 3.136 ppm. The signals of the carbon-coupled AR-H hydrogen in the aromatic ring are triplet at 7.492 ppm and 7.377 ppm, double signals at 7.610 ppm and 7.808 ppm. Based on the signals in the benzene ring, we can conclude that the reactant product is oriented towards the Ortho state rather than the para state with respect to the n, n-dimethyl group.

In a ¹³C NMR analysis of N, N-dimethyl(amino) phenacyl chloride at C¹³, it can be seen that N-44.941 and 46.684 were hybridized and signalized at ppm fields, which formed a bond with the nitrogen atom sp³. Multiplet signals can be observed at 120, 122, 128, 129, 138 and 144 ppm at the expense of carbon fluctuations in the AR-C bond in the aromatic ring. we can see that the Cl-C-H bond in the methylene group that formed a bond with the sp³ hybridized chlorine atom is signalized at Carbon 39.9 ppm. The carbon of the AR-C=O bond in the carbonyl group attached to the aromatic ring showed a signal at a field of 95.5 ppm.

LIST OF USED LITERATURE

1. Abbas Q., Ashraf Z., Hassan M., Nadeem H., Latif M., Afzal S., Seo S.-Y. ' Development of highly potent melanogenesis inhibitor by in vitro, in vivo and computational studies // Drug Design, Development and Therapy, -2017. -V. 11 -P. 2029-2046.
2. Rajesh Kumara, Maninder Kaur, Malkeet Singh Bahia, Om Silakari. Synthesis, cytotoxic study and docking based multidrug resistance modulator potential analysis of 2-(9-oxoacridin-10(9H)-yl)-N-phenyl acetamides // European Journal of Medicinal Chemistry - 2014. -№. 80. -P. 83-91.
3. Mamatkulov N. N., Ochilov M., Tursunova D. R. 5- Methyl -2-(1-Methylethyl) phenol with chloroacetyl chloride under alkaline conditions//Web of Semantic: Universal Journal on Ie Education //Volume 2 Issue 4, Year 2023 ISSN: 2835-3048 <https://univerpubl.com/index.php>.
4. Mamatkulov N.N. Chemical Treatment of Water In Ammophos Production Plants// The American Journal of Agriculture and Boimedical Engineering (ISSN – 2689-1018) Published: June 18, 2021.
5. Маматкулов А. К. Абдушукуров., Ш. Хидиров., С. Рахмонова. Получение и перегруппировка п-толилового эфира хлоруксусной кислоты// Журнал Органической химии. Т. 37. вып. 11. -Санкт-петербург, «Наука». 2001. -С. 1738-1739.
6. N.N. Mamatkulov. Superfos o'g'it ishlab chiqarishda klassifikator qurilmasining hisobi// Oriental Renaissance: Innovative, educational, natural and social sciences. volume 1 | issue 5 issn 2181-1784. Scientific Journal Impact Factor. SJIF 2021: 5.423.