WEB OF SYNERGY:
International Interdisciplinary Research Journal
Volume 2 Issue 4, Year 2023 ISSN: 2835-3013
https://univerpubl.com/index.php/synergy

2-Isopropyl-5-Methylphenylchloroacetate Synthesis Method

Abdushukurov Anvar Kabirovich
Professor 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

Ibragimov Takhirbek Egamshukurovich
Teacher of the Faculty of Chemical Technology of Inorganic Substances, Tashkent Institute of Chemical Technology

Jadilova Dilnavoz Abdulazizovna
Student of the Faculty of Chemistry of the University of Technical Uzbekistan

Article Information

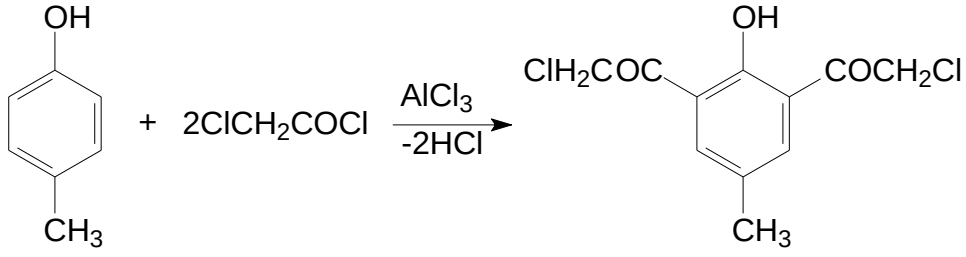
Received: February 18,2023
Accepted: March 19,2023
Published: April 18,2023

Keywords *Chloroacetyl chloride, phenol, cresol, catalyst, benzene, solvent, extraction, vacuum, flask, sodium metal, O-acylation, S-acylation, inglet, mixture, 1,4- benzodioxane*

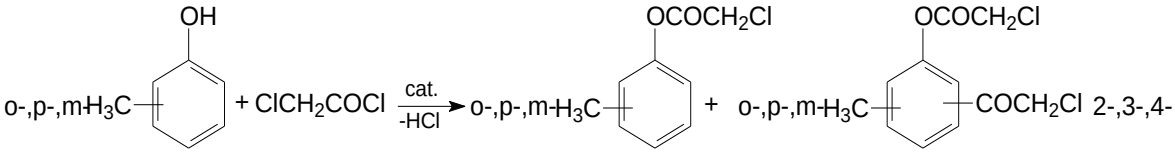
ABSTRACT

In this article, the reaction of 2-isopropyl-5-methylphenol with chloroacetyl chloride with benzene and 1,4-benzodioxane was carried out for the first time. It was found that the yield of the substance in this reaction depends on the solvent and the reaction conditions. It was observed that the reaction of 2-isopropyl-5-methylphenol with sodium metal leads to chloroacetylation of 2-isopropyl-5-methylphenolate. The structure of the new substance formed was confirmed by IR-, H^1 CMR and C ^{13} CMR spectra.

It is known from the literature that when p-cresol is chloroacetylated in the presence of a large amount of $AlCl_3$, the monoacyl product 2-hydroxy-5-methylphenacyl chloride is formed. If the amount of catalyst is doubled and the diethyl product 2,6-dichloroacetate-4-methylphenol is formed, which is 26% reached [1-2].

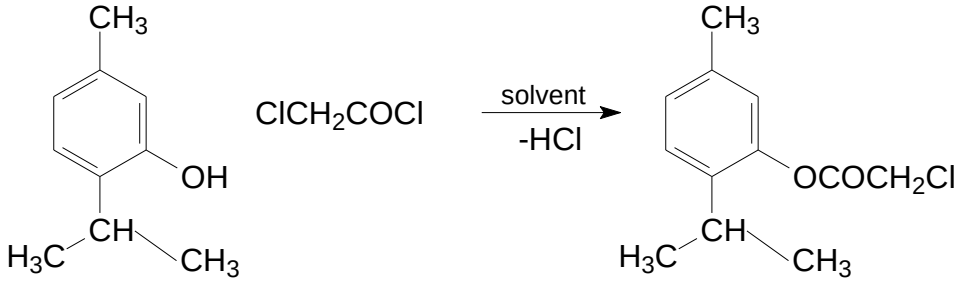


Phenol and o-, m- and p-cresols were reacted with chloroacetyl chloride in the presence of catalytic amounts of $FeCl_3$, $FeCl_3$ $06N_2O$, $ZpCl_2$, $Fe_2(SO_4)_3$ and TAA catalysts. As a result of the reaction, the O-acylation reaction took place, and phenyl- and o-, m- and p-tolyl chloroacetates were formed. S-acylation of the aromatic ring leads to the formation of various isomers [3-4].

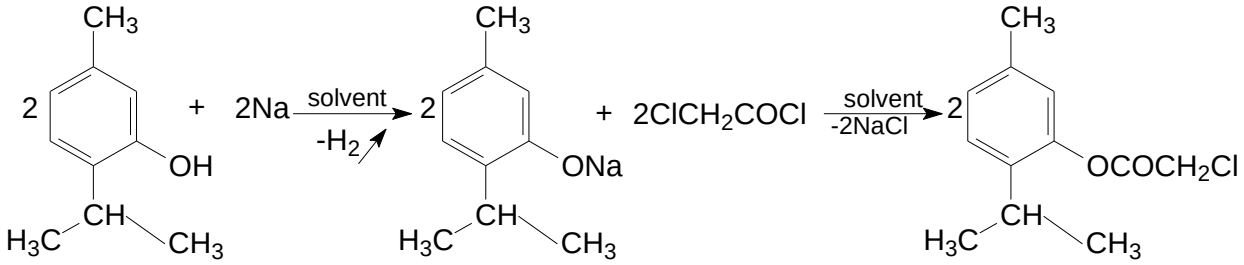


These scientists found that only the O-acid product is formed during the chloroacetylation reaction of phenol and isomer cresols in benzene solution.

The reaction of 2-isopropyl-5-methylphenol, benzene and chloroacetyl chloride in a solution of 1,4benzodioxane was studied during the synthesis of new organic substances. As a result of the reaction, it was determined that one substance in the individual state is formed.



As a result of the reaction it was found that 2-isopropyl-5-methylphenol chloroacetylation reaction is carried out in the presence of sodium metal, and the above-mentioned substance is formed. Thin layer chromatography of the substance revealed the presence of a single substance. The structure of the isolated substance was confirmed by IR and PMR spectra. The reaction goes according to the following scheme.



The structure of the isolated substance was confirmed by IR and CMR spectra.

Experience part

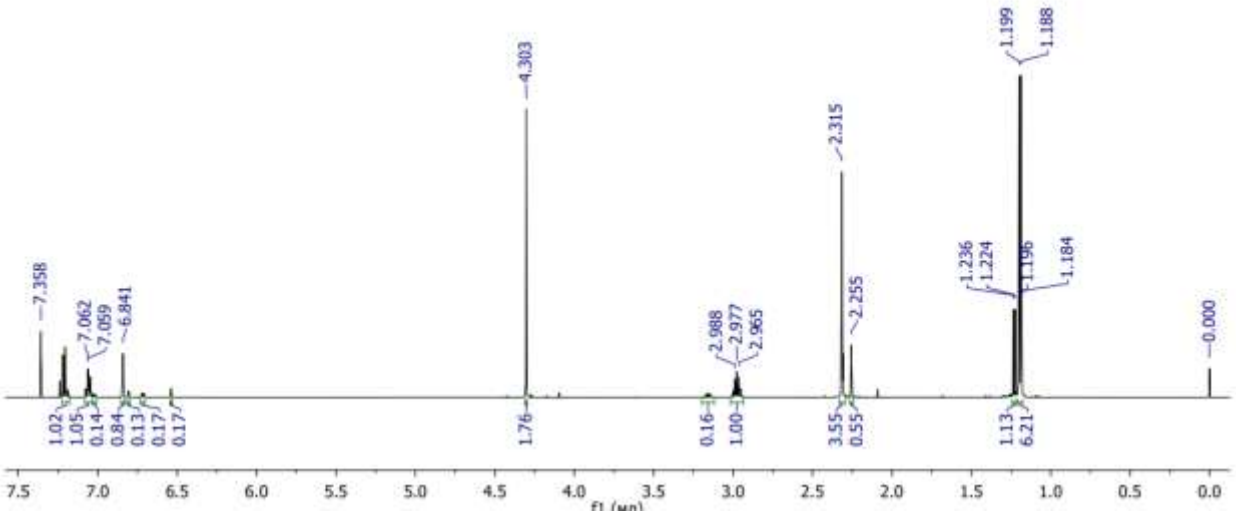
Experience №1. 11.32 g (0.05 g-mol) of 2-isopropyl-5-methylphenol, 50 ml of abs. dissolved in benzene, 5.65 g (0.05 g-mol) of chloroacetyl chloride was added and boiled for 18 hours. The completion of the reaction was determined by the cessation of hydrogen chloride evolution. The mixture was then washed with 10% aqueous alkali, extracted with benzene, and dried over $CaCl_2$. Benzene is simple in circumstances , mod while in vacuum 1 40 -1 45 ^{0} C /20 mm. wire he kicked out. of the resulting 2-isopropyl-5-methylphenylchloroacetate product 7 g (62 %).

Experiment №2. 11.32 g (0.05 g-mol) of 2-isopropyl-5-methylphenol was dissolved in 60 ml of 1,4-benzodioxane in a round-bottomed flask fitted with a tube adapted for the release of

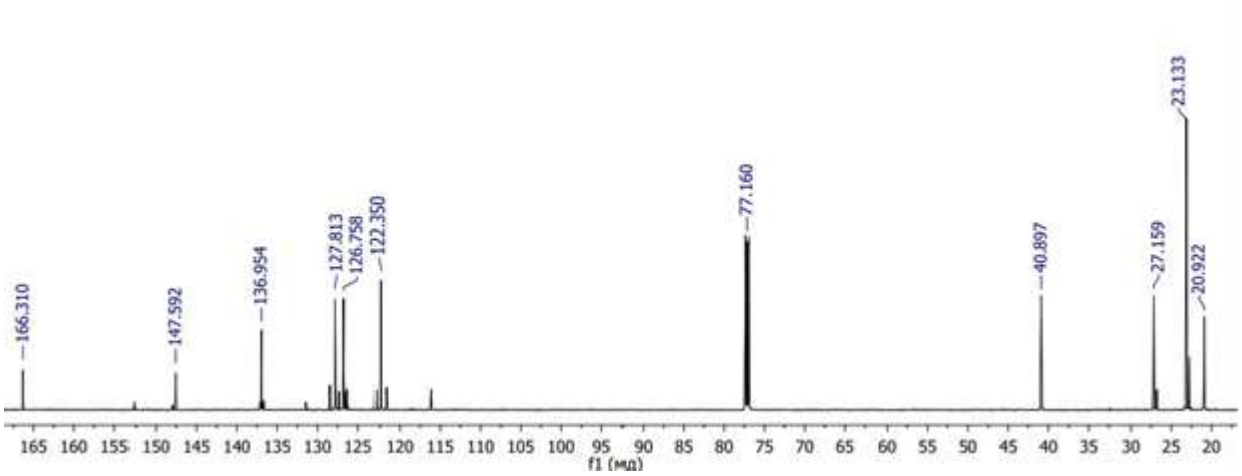
hydrogen chloride to a reflux condenser, 5.65 g (0. HYPERLINK "https://chemister.ru/Database/properties-en.php?dbid=1&id=146" 05 g-mol) of chloroacetyl chloride was added and boiled for 7 hours. After the evolution of hydrogen chloride stopped, the mixture was washed with 10% alkaline water. Extracted in benzene and dried over $CaCl_2$. Benzene is simple in circumstances , mod while in vacuum 1 40 -1 45 ^{0} C /20 mm. wire he kicked out. of the resulting 2-isopropyl-5-methylphenylchloroacetate product 8.26 g (7 %).

Experiment №3. 11.32 g (0.05 g-mol) of 2- isopropyl-5-methylphenol was dissolved in 60 ml of 1,4-benzodioxane in a round-bottomed flask equipped with a tube adapted for the release of hydrogen chloride to the reflux condenser. Little by little on him 1, 15 g (0.0 5 g-atom) of the oxide film purified sodium metal was added. After the formation of sodium 2-isopropyl-5-methylpheniate slowed down, the reaction mixture was heated at the reflux temperature of 1,4-benzodioxane for 4 hours. Then, 5.65 g (0.05 g-mol) chloroacetyl chloride was added slowly and the reaction was carried out for 3 hours. After completion of the reaction, 1,4-benzodioxane was removed under normal conditions and the mixture was washed with 10% alkaline water. The product was extracted in benzene and dried over $CaCl_2$. Benzene under normal conditions, and 2-isopropyl-5-methylphenylchloroacetate under vacuum at 140-145 ^{0} C /20 mm. sim. above kicked out. The yield is 9.7 g (86%).

Spectral analysis of the obtained reactions



In the H^1 CMR analysis of 2-Isopropyl-5-methylphenylchloroacetate, a singlet signal at 2.977 ppm (mu) was observed for the Ar-CH_3 bond of the methyl group sp^3 hybridized and bonded to the benzene ring. We can observe that sp^3 hybridized and hydrogen in the tertiary carbon R_3 -CH group recorded a quartet signal in the range of 1.184-1.236 ppm (mu) . Aromatic in the ring to carbon connected Ar -H of hydrogen signals at 7.168 ppm and at 7.062 pmm (mu). doublet , singlet signals at 7.358 ppm (mu). observed . In Y on chains simple air garden and acyl group held of carbon hydrogen ROC H-COCl 4.303 ppm (mu) showing a singlet signal in the field did.



In the C^{13} NMR analysis of 2-Isopropyl-5-methylphenylchloroacetate, it can be seen that it exhibits signals in the 20.9, 23.1 and 27.1 ppm (mu) regions due to the vibration of the carbon in the sp^3 hybridized -CH_3 group . sp3 hybridized and tertiary of carbon vibration due to R3-CH signal at 40.897 ppm (mu) was observed. Aromatic in the ring in the Ar-C bond of carbon vibration at 122.3, 126.7, 127.8, 136.9, 147.5 and 166.3 ppm (mu) multiplet signals observation can Atsil in the group chlorine atom garden with did carbon COCl garden that a signal was observed at carbon 77.160 ppm (mu). Let's see can

of 2-Isopropyl-5-methylphenylchloroacetate IR spectra were taken on a Bruker INVENIO-S Fure spectrometer manufactured in 2021 (4000-400 cm^{-1} , ATR).Received IR of 2 -isopropyl-5- methylphenol in the spectrum 303 Absorption lines of the Ar - H bond bonded to the sp^2 hybridized C atom of the aromatic ring at 3 cm^{-1} , 17 absorption lines formed by the valence vibrations of the C = O bond in the 60 cm^{-1} region , and 29 6 2 -287 1 cm^{-1} absorption lines of the C - H bond bonded to the sp^3 hybridized C atom were observed in the field gap. - SN_3 valence vibration of group 1457 cm^{-1} field showed absorption lines .1.2 and 1.5 cm^{-1} in areas

728-726 and 812-868 cm^{-1} showed absorption lines in the spheres .

LIST OF USED LITERATURE

1. Cullinane NM, Edwards FR Comparison of the Fries and Friedel-Crafts reactions //J.Appl. Chem. -1959. - Vol. 9. -P.133-136.
2. Fries K., Fink G. uber Homologe des cumarons und itheabkemmlinge // Ber. 1908. B. 41. S. 4271-4284.
3. Mamatkulov N.N. V liyanie sootnoshenia reagentov i katalystara na vykhod reaktsii chloracetilirovaniya phenol i isomernykh crezolov// UNIVERSUM: Teknicheskiy nauki .N auchnyy zurnal.Vypusk 4 (82) . April 2021. Moscow. 77-80 -S.
4. Mamatkulov NN Development of the mechanism of action and reaction of O-chloromethylation // International Journal of advanced Research in Science, Engineering and Technology. India. Vol. 7 . Issue 1, January 20 20 . Pp. 12636 – 12639.
5. N.K. Madusmanova Determination of iron ions from environmental objects // Oriental renaissance: Innovative, educational, natural and social sciences 2021. 1.5.C. 230-233.