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ORGANIC MAGNESIUM COMPOUNDS SYNTHESIS OF AROMATIC ACETYLENE ALCOHOLS

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ОРГАНИЧЕСКОЕ МАГНИЕВОЕ СОЕДИНЕНИЕ СИНТЕЗ АРОМАТИЧЕСКИХ АЦЕТИЛЕНОВЫХ СПИРТОВ

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ABSTRACT

Aromatic acetylenic alcohols (AAA) were synthesized as a result of the reaction of methyl propyl ketone, diethyl ketone, methyl isopropyl ketone and pinocoline with phenylacetylene in the presence of a Grignard reagent. The effect of DEE and THF solvents on the product yield was studied. It was determined that the average yield of AAA is 11.0 % higher in THF than DEE.

АННОТАЦИЯ

В результате реакции метилпропилкетона, диэтилкетона, метилизопропилкетона и пиноколина с фенилацетиленом с реактивом Гриньяра синтезированы ароматические ацетиленовые спирты (ААС). Изучено влияние на выход продукта растворителей ДЭЭ и ТГФ. Определено, что выход ААС в средном на 11,0% больше в растворителе ТГФ, чем ДЭЭ.

Keywords: ketones, methylpropyl ketone, diethyl ketone, methyl isopropyl ketone, pinocoline, phenylacetylene, organic magnesium, phenylacetylene, aromatic asetylenic alcohols.

Ключевые слова: кетоны, метилпропилкетон, диэтилкетон, метилизопропилкетон, пиноколин, фенилацетилен, магний органическое соединение, фенилацетилен, ароматические ацетиленовые спирты.

Introduction. The role of elemental organic compounds is important in organic chemistry. Currently, in organic synthesis, various new compounds are being synthesized based on elemental organic compounds.

Metal organic compounds were first synthesized by A.D. Franklin in 1849 [1]. In 1829 Sh.R. Barbe synthesized mixed metal organic compounds [2]. The French scientist V.M. Grignard synthesized mixed magnesium organic compounds (Grignard's reagent) by reacting powdered magnesium metal to a solution of alkyl halides in dehydrated ether (Iotsich reagent) [3-4]. Based on this, it is called the Grignard-Iotsich method [5].

Organometallic reagents are often used in the synthesis of organic molecules because they promote the formation of certain bonds and (or) catalyze the reaction. Individual reactions are difficult or impractical to carry out otherwise. In most organic compounds, the carbon atoms are electrophilic, but in organometallic compounds, the metal atom usually has less electronegativity than its bonded carbon atom, so the carbon becomes nucleophilic with varying strength. When a metal with high electronegativity is used, the charge distribution increases the intensity of its ionic properties and the reactivity of the compound [6-7]. New stable compounds were synthesized by Iotsich on the basis of reaction of Grignard's reagent with alkynes [8-10].

In this research work, aromatic acetylene alcohols (AAA) were synthesized based on the methods presented in the literature. In order to determine the range of relative efficiency of the synthesized

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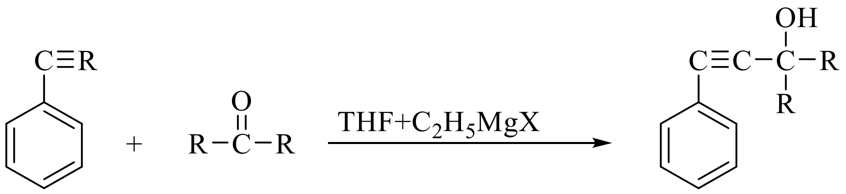
compounds, their reactions based on organic metal compounds were also studied. Studies were conducted on the process of synthesis of AAA on the basis of organic magnesium compounds used on a large scale in organic synthesis, according to the Grignard-Iotsich reaction. Based on the reaction of ketones - methylpropylketone, diethylketone, methylisopropylketone, pinokalins- with magnesium organic compounds based on Grignard-Iotsich reaction, corresponding AAA was synthesized. The reaction equation was proposed as follows.



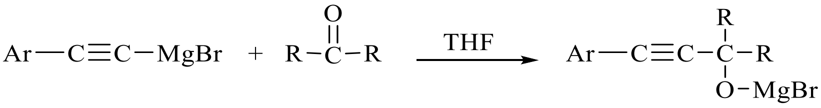
Here it is: R = $-CH_3$, $-C_2H_5$, $-C_3H_7$, iso- C_3H_7
X = halogen

Reaction to obtain aromatic acetylic alcohols

The formation of the iodine complex is due to the active hydrogen in phenylacetylene. In this case, phenylacetene was added dropping to a solution of ethylene ethyl magnesium bromide (Grignard's reagent) in TGF, and the magnesium bromide derivative of phenyl acetylene (Iotsich's reagent) was prepared.

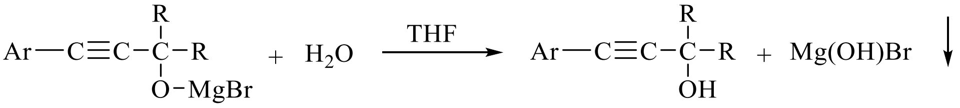
$$Ar{-}C{\equiv}CH + C_2H_5MgBr \xrightarrow{THF} Ar{-}C{\equiv}C{-}MgBr + C_2H_6\uparrow$$

The resulting Iotsich reagent reacts with ketones to form intermediate AAA metal halide salts.



$$Ar{-}C{\equiv}C{-}MgBr + R{-}\overset{\overset{O}{\|}}{C}{-}R \xrightarrow{THF} Ar{-}C{\equiv}C{-}\underset{O-MgBr}{\overset{R}{C}}{-}R$$

AAA was formed when the resulting substance was hydrolyzed in ice water.



$$Ar{-}C{\equiv}C{-}\underset{O-MgBr}{\overset{R}{C}}{-}R + H_2O \xrightarrow{THF} Ar{-}C{\equiv}C{-}\underset{OH}{\overset{R}{C}}{-}R + Mg(OH)Br\downarrow$$

Experimental part. A sieve, a reflux condenser, a thermometer and a dropping funnel are installed in a four-necked flask and 75 ml of absolute tetrahydrofuran and 3.05 g (0.125 mol) of powdered magnesium metal are mixed in it. Then, through the dropping funnel, 19.0 g of ethyl bromide and equal amount of tetrahydrofuran mixture is added. The reaction mixture is boiled vigorously for 50-55 minutes. The resulting product (Grignar reagent) was cooled to room temperature (25-27 °C) and stirred for another 60 minutes.

12.5 g (0.125 mol) of phenylacetylene is dripped with stirring to the Grignard reagent solution formed as a result of the reaction for 2 hours (the temperature of -5-0°C in the flask is created by liquid nitrogen), then an equal amount of absolute ether solution of 0.25 mol of ketones is added to it over 2 hours. Upon completion of the reaction, the resulting mixture (3x25 ml) was hydrolyzed with ice water and diluted hydrochloric acid was added until the precipitate dissolved. After separating the part of organic substances, the aqueous part is extracted again (3x25 ml). The extract is dried with sodium sulfate or potash, the diethyl ether is driven off under normal conditions, and the remainder is fractionated by driving under vacuum.

Research results and analysis. Depending on the structure of the selected ketones, different AAA are formed as a result of the reaction with phenylacetylene. Including 25.5 g (0.25 mol) of phenylacetylene and 21.5 g (0.25 mol) of methylpropylketone 3-methyl-1-phenylhexin-1-ol-3 (1) (80.0 %) nD 1.5126, d_2=1.1261 g/cm^3; Using 25.5 g (0.25 mol) of phenylacetylene and 21.5 g (0.25 mol) of diethyl ketone, 3-ethyl-1-phenylpentin-1-ol-3 (2) (79.3 %), nD=1,5333, d_2=1.0765 g/cm^3 ; 25.5 g (0.25 mol) of phenylacetylene and 21.5 g (0.25 mol) of 3,4-dimethyl-1-phenylpentin-1-ol-3 using isopropyl ketone (3) (76.4%) p_2=1.5400, d_2=1.0010 g/cm^3; From the reaction of 25.5 g (0.25 mol) of phenylacetylene and 25.0 g (0.25 mol) of pinokalin, 3,4,4-trimethyl-1-phenylpentin-1-ol-3 (4) (72.1 %), *n*™ = 1.5360, d_2 =1.9223 g/cm^3 were synthesized.