

SYNTHESIS OF 9-DECEN-1-OL

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Abstract: Considering that this paper deals with alcohols, we will point out their most important characteristics. We will follow the order known in the organic chemistry. Alcohols are divided into three large groups:

- Primary alcohols,
- Sterile alcohols,
- Carbohydrates.

Considering that we are interested in basic alcohols and the alcohols difficult to synthesize industrially, we will show simply and in the broadest terms some of the characteristics of all alcohols, as well as of unsaturated alcohols.

Keywords: Alcohols, phenols, Organic chemistry

Obtaining alcohol from unsaturated
carbohydrates

Very often the synthesis of alcohols uses alkenes that react with mercury acetate – oxymercuration, and gives organo-metal compounds in large amounts, which reduction from NaBH creates 80% alcohols. Oxy mercury and dimercury represent the alkene hydration according to the Markovnikov’s rule with minimal side products and reactions.

INTRODUCTION

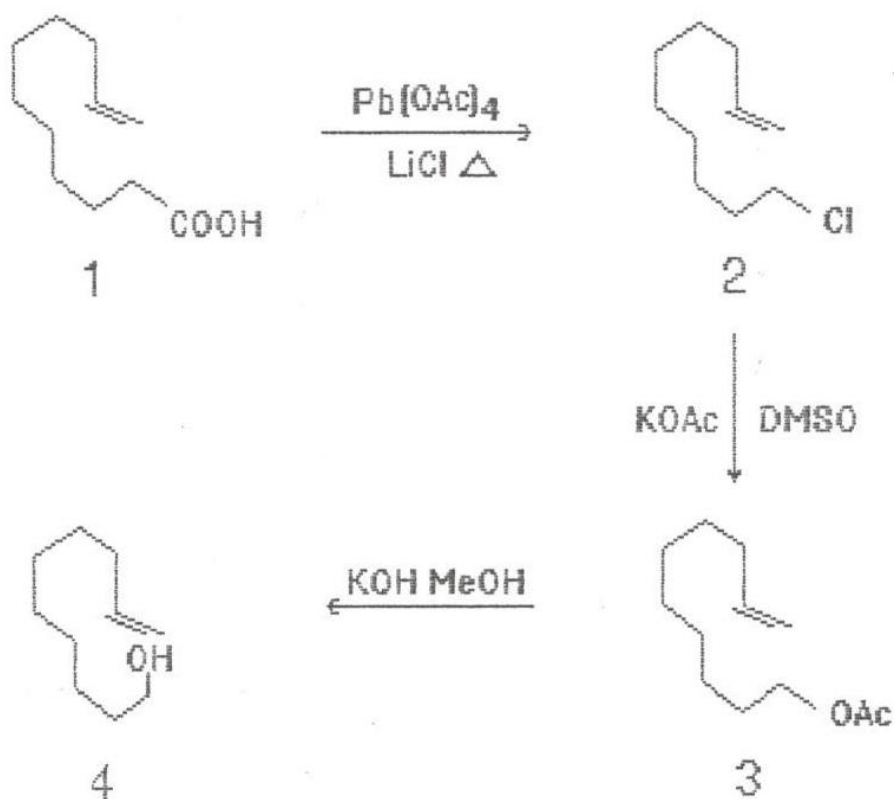
A hydroxyl group –OH manifests as a functional group in two types of compounds, alcohols^{1,2,3} and phenols. In case of phenols, it is bonded to the sp²-hybridized carbon atom of benzene system and therefore it shows certain chemical properties. Saturated alcohols contain the –OH group bonded with sp³-hybridized carbon atom. Apart from these compounds containing the –OH group, there are also other compounds which may contain the –OH group that is not stable and is subject to changes (enols).

OUR WORKS

I Synthesis of 9-decenol

I₁ Synthesis of 9-decenol (4)⁴ from undecylenic acid

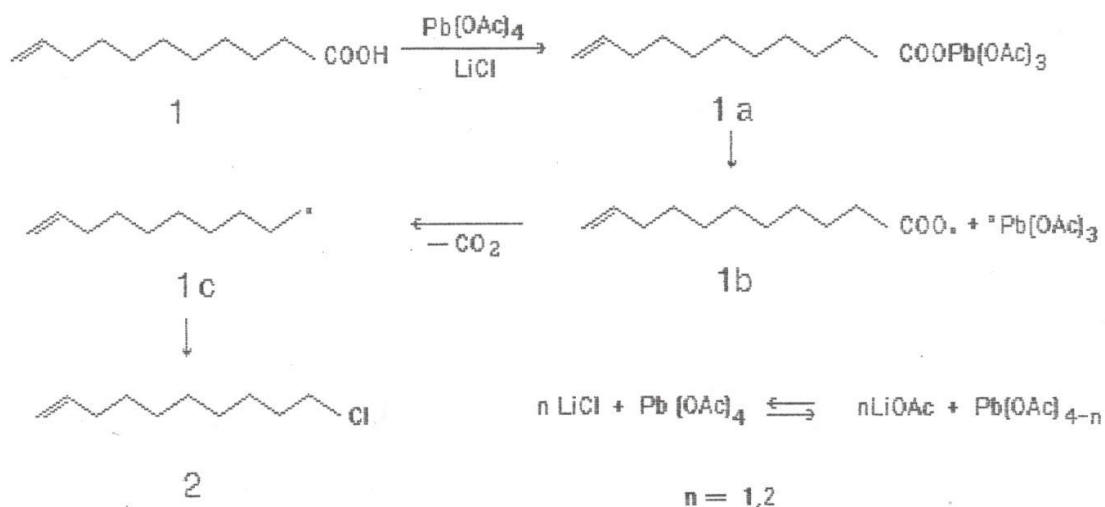
The synthesis of 9-decenol (4) was conducted in three phases, and undecylenic acid (1) was used as initial substance in this synthetic procedure. The key phase in this synthesis is the oxidative decarboxylation of undecylenic acid (1) up to 9-decyl-chloride (2), as shown on the scheme 1⁴



SCHEME - 1

Oxidative decarboxylation of undecylenic acid was completed with the lead of tetra acetate. However, instead of the cupric acetate, as electron-transfer reagents, these oxidations were performed in the presence

of LiCl, which enables the transfer of chloride ion as ligand. This type of oxidative decarboxylation is known as halo-decarboxylation, scheme 2^{5,6}



SCHEME - 2

This decarboxylation also contains the oxidation of alkene radicals (iC) with help of mixed lead salts from which the atom of Chlorine is taken and 9-decynil chloride is made (2),

The stabilization of alkene radicals (i-c) is also present in other radical or oxidative processes, which leads to the creation of side products (acetate esters, unsaturated carbohydrates, etc.)

General notes

Fractional distillation under reduced pressure was performed with modified well-isolated Vigreux column.

Quantitative determinations were performed with gas chromatography by using Varian-Aerograph apparatus (equipped with flame ionization detector).

Metal columns were used (2m x 2mm) with different charging. The column temperature, detectors sensitivity, carrier gas flow, and the concentration of liquid phase in the column are adjusted according to the physical and chemical characteristics of the analyzed substance. Argon is used as the carrier gas.

Preparatory separations were performed on Varian-Aerograph apparatus with different charging.

IR was performed with Perkin-Elmer intracortic apparatus of different models.

The substances were recorded as a film or in the gas carbon-tetra chloride.

The NMR specter was recorded on the apparatus Varian F-T-80 and Varian A-60-a.

EXPERIMENTAL PART

II Synthesis of 9-decenol (4)^{4,5}

II_{2.1}. Synthesis of 9-decenil-chloride

In the three-necked flask of 50ml, equipped with reverse condenser, a device for argon introduction and a mixer, 4.6g (0.025 mol) of undecylenic acid is placed, or 11g (0.02 mol) of lead tetra-acetate, 1.06g (0.0025 mol) of lithium chloride and 30ml of anhydrous benzene.

The mixture is mixed in the argon atmosphere at 80°C. After the completion of the reaction, the solution is filtered, rinsed with water, and then with saturated solution of sodium bicarbonate and with water again, until the reaction becomes neutral. The same solution is left to dry over the anhydrous magnesium sulfate. After the distillation separation of benzene at the atmosphere pressure, the rest of the reaction mixture is distilled at 85°C under the pressure of 933.256 Pa. The 9-deceny-chloride obtained in this mixture is 3.5g (80%).

II_{2.2}. Synthesis of 9-deceny-acetate (3)^{4,5}

In the two-necked flask of 50ml, equipped with reverse condenser, a device for argon introduction and a magnetic mixer, 1.04g (0.0059 mol) of 9-deceny-chloride is placed (2), 1.171g (0.0118 mol) of potassium-acetate and 10ml of dimethyl-sulfoxide. The mixture is heated and stirred at 130-140°C for the duration of 1.5-2 hours. After the completion of the reaction, the organic layer containing the 9-deceny-acetate is rinsed with water and

the solution of sodium bicarbonate until neutral reaction, and then is left to dry with anhydrous sodium-sulfate.

Upon separating the dimethyl-sulfoxide with distillation under normal pressure, the rest of the reaction is distilled at the temperature of 136°C under the pressure of 799.934 Pa. The obtained 9-decenyl-acetate (3) is 0.52g (44%) in comparison to the initial 9-decenyl-chloride.

I_{2.3}. Synthesis of 9-decenol (4)^{4,5}

In a round flask of 50ml, equipped with reverse condenser and a magnetic mixer, 1.9g (0.01mol) of 9-decenyl-acetate is placed, 0.56g (0.01 mol) of potassium-hydroxide, 3ml H₂O and 2ml of methanol. The mixture is heated and left to boil for 1-2 hours on the oil bath. After the completion of the reaction, the solution is cooled down, 3ml of water is added and then it is acidified with diluted hydrochloric acid. The organic layer is extracted with ether and left to dry with anhydrous sodium-sulfate. Ether and methanol are separated with normal pressure distillation, and the rest of the

solution is distilled under the lowered pressure. Unsaturated alcohol, 9-decenol (4) is distilled at T=155°C or at 120° under the pressure of 533.289 Pa.

The obtained 9-decenol is 1.575g (86%) in comparison to the used ether, that is 30.48% in comparison to the initial undecylenic acid.

STRUCTURES OF SYNTHETIC PRODUCTS

The structure of initial substrata and the reaction products are determined on the basis of infrared specters and specter proton nuclear magnetic resonance. On the basis of these data, the reaction compounds are characterized. The liquid compounds are purified with fractional distillation and/or preparatory gas chromatography. This chapter lists the specter data on the basis of which the compound identification was performed.

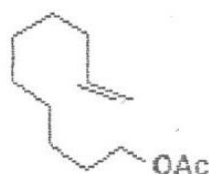
9-decenyl-chloride (2)



IR (film) δ cm^{-1} : 3080,2930,1860,1640,
1460,1360,995,910,720,650

9-decenyl-acetate

(3)



IR (film) δ cm^{-1} : 3080,2970,2850,1735
1640,1460,1420,1360,1310,1230,1040
910,905

CONCLUSION

On the basis of the results obtained from the studied reactions, the following conclusions may be drawn:

1. The synthesis of 9-decenol (4) was performed in three phases by using the undecylenic acid (1) as initial substance.
2. The main phase in this synthesis is the reaction of decarboxylation of undecylenic acid (1) with lead-tetra acetate in the presence of lithium-chloride up to 9-deceny chloride with the yield of 3.5g (80%) in comparison to the initial substance.
3. The total amount of the obtained 9-decenol is 1.575g (86%) in comparison to the initial substance.

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