The effect of various factors on the yield of 2,3,3–triiode propene–2–ol–1

Gulrux Khaidarova1*, Abdukhamid Makhsumov2, Dilshod Akhtamov1, and Lola Tilavova1

1Navoi State University of Mining and Technologies, Navoi, Uzbekistan
2Tashkent Institute of Chemical Technologies, Tashkent, Uzbekistan

Abstract. A triode derivative of propargyl alcohol has been synthesized, and the effects of catalyst concentration, time and temperature on the synthesis process have been studied. Various concentrations of alkalis (5.0–30.0 mass. %) have been used as catalysts. It has been found that 2,3,3–triiode propene–2–ol–1 has a higher yield when the concentration of the catalyst NaOH is 15%. The effect of time duration (1–5 hours) on product productivity has also been studied. Product yield has been found to be high for 3 hours. In addition, the effect of temperature on the product's productivity has also been studied at intervals of (10–60°C). It has been found that the yield of 2,3,3–triiodepropen–2–ol–1 was high in 3 hours at 20°C. The structure of the synthesized substance was studied and confirmed by the methods of IR– and NMR–spectroscopic analysis.

1 Introduction

Unsaturated alcohols and their derivatives are important among organic synthesis products. Including taking its halogen derivatives on the basis of propargyl alcohol is currently paid a lot in the world [1]. These compounds are multifunctional in nature and are also widely used in many fields, including pharmaceutical as fungicide, bactericide and antimicrobial agent, agricultural as herbicides, industrial as corrosion inhibitors, in addition to being antipyretics for polymers materials [2–5].

Among the halogen derivatives of propargyl alcohol, the most significant are propargyl bromide, propargyl chloride, 2,3,3–triiode propene–2–ol–1, 3–bromine 2,3–diode propene–2–ol–1, 3–iodine propine–2–ol–1, and 3–bromine propine–2–ol–1 [6-11].

The study carried out the synthesis of 2,3,3–triiode propene–2–ol–1 on the basis of propargyl alcohol and iodine crystals, and studied the nature and concentration of catalysts, temperature, the effect of reaction time on the product yield. In the course of the study, many techniques were used for the synthesis of 2,3,3–triiode propene–2–ol–1.

2 Materials and methods

Research objects are propargyl alcohol (Tl= 113.6 °C, d= 0.9485 g/sm³, n²₀= 1.4322), NaOH (Tl = 1403 °C, d= 2.13 g/sm³, n²₀= 1.457), KOH (Tl = 1325 °C, d= 2.044 g/sm³, n²₀= 1.48)

* Corresponding author: dilshod.axtamov.89@mail.ru

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LiOH (Ti = 925 °C, d = 1.46 g/sm³, n^20_D = 1.460), RbOH (Ti = 1390 °C, D = 3.203 g/sm³, n^20_D = 1.489) and acetone (Ti = 56.1 °C, d = 0.7899 g/sm³, n^20_D = 1.3588).

The structure of the synthesized compound has been studied and confirmed by IR spectroscopic, ^1H and ^13C NMR spectroscopic methods. NMR–spectra were recorded in JNM–ECZ400R spectrometer (Jeol, Japan) for ^1H and ^13C in CDCl3 and CCl4 solution at a working frequency of 600 MHz.

Synthesis of 2,3,3–triode propene–2 ol–1: 2,3,3–triode propene–2 ol–1 is directly exposed to the propargyl alcohol by iodine crystal in alkali catalysis (LiOH, NaOH, KOH, RbOH). 2.8 g of propargyl alcohol and 60 ml of water are poured into a three–neck flask and 25.4 g of iodine is added while stirring, then a 15% solution of NaOH as a catalyst is infused for 30 minutes. The reaction is carried out at 20°C for 3 hours. The resulting precipitate is separated, the resulting product is first washed and dried using a 10% solution of Na2S2O3, and then in water. The dried product is recrystallized in acetone. The 2,3,3–triode propene–2–ol–1 (Ti=151–1520C) obtained after the reaction was found to have a yield (16.05 g) of 73.62%.

3 Results

The study investigated the effects of 2,3,3 triiodes propene–2–ol–1 synthesized under different reaction conditions and various factors on productivity: catalyst nature and concentration, temperature and time.

The research process is based on the following reaction equation:

\[
\text{H}_2\text{C} \equiv \text{C} \equiv \text{CH} + \text{I}_2 \xrightarrow{\text{Cat.} \ 3 \ h \ 20^\circ\text{C}} \text{H}_2\text{C} \equiv \text{C} \equiv \text{I} + \text{HI}
\]

Cat.: LiOH, KOH, NaOH, RbOH

The effect of temperature on the fertility of 2,3,3–triode propene–2–ol–1 was studied in the 10–60°C temperature range, and the results obtained are shown in Table 1. Analysis of the results of the study shows (Table 1) that the optimal temperature for the output yield of the main product is 20°C. Further increase in temperature leads to a decrease in the productivity of the main product. This can be explained by the easy sublimation of iodine from the solution content at high temperatures.

<table>
<thead>
<tr>
<th>Structure formula and name of the substance</th>
<th>Temperature, °C</th>
<th>Temperature, °C</th>
<th>Temperature, °C</th>
<th>Temperature, °C</th>
<th>Temperature, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10</td>
<td>20</td>
<td>30</td>
<td>40</td>
<td>50</td>
</tr>
<tr>
<td>HOH2C–C≡C≡I</td>
<td>56.75</td>
<td>73.39</td>
<td>70.16</td>
<td>67.59</td>
<td>60.39</td>
</tr>
<tr>
<td>2,3,3–triode propene–2–ol–1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The influence of the nature of the catalyst on the product productivity was also studied in the research process, and its results are presented in Table 2.
Table 2. The effect of the catalysts’ nature on the yield of 2,3,3–triiodo propene–2–ol–1.

<table>
<thead>
<tr>
<th>Structure formula and name of the substance</th>
<th>Catalysts</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>LiOH</td>
</tr>
<tr>
<td>2,3,3–triiodo propene–2–ol–1</td>
<td>47.31</td>
</tr>
</tbody>
</table>

Analysis of the results showed that among the catalysts tested by the research carried out, NaOH was found to have an effective effect on the productivity of products. This can be explained by the increase in the basicity properties of catalysts.

In reactions, the amount of Catalyst has a huge impact on the productivity of products. Therefore, the effect of the concentration of catalysts, which effectively affected the productivity of products, was also studied. In this case, the effect of the NaOH catalyst concentration, which effectively affects the productivity of products among catalysts, was studied in detail, the results of which are presented in Figure 1 below.

![Fig. 1. The effect of catalyst concentration on yield 2,3,3–triiodo propene–2–ol–1: 1) NaOH; 2) KOH; 3) LiOH; 4) RbOH.](image)

Analysis of the results showed that the yield of the products formed also increased when the catalyst concentration used in the research processes increased by 5–15%. It was found that the yield of products decreased significantly when the concentration of the catalyst increased by 20–30%. This is due to the fact that more than 15% of the catalyst reacts with the initially interacting halogen, that is, affects the equivalent amount of halogen, which also affects the productivity of self–generated products. The highest yield of the resulting product was observed when a 15% concentrated solution of NaOH catalyst was used, and the yield of 2,3,3–triiodo propene–2–ol–1 was 73.6 %.

Another of the factors that affect the fertility of the halogenation reaction of propargyl alcohol is the duration of the reaction. The time correlation of the yield of 2,3,3–triiodo propene–2–ol–1 is given in Table 3.
Table 3. Effect of reaction time on the yield of 2,3,3–triiode propene–2–ol–1.

<table>
<thead>
<tr>
<th>Structure formula and name of the substance</th>
<th>Time, (hour)</th>
<th>Yield, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>HOH₂C≡C≡C—I</td>
<td>1</td>
<td>63.35</td>
</tr>
<tr>
<td>2,3,3–triiode propene–2–ol–1</td>
<td>2</td>
<td>68.88</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>73.39</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>67.85</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>61.37</td>
</tr>
</tbody>
</table>

From the analysis of the results (Table 3), it can be seen that yield increased in stages when the reaction duration increased to 3 hours, and the highest rate reached 73.39%, while when the duration of time was further increased, it was observed that the yield decreased.

This can be explained by the fact that the double bond product has become oligomeric and polymer products.

The structure of the initial and synthesized substances was studied using methods of Physic–Chemical analysis, namely IR– and NMR–spectroscopic methods. The IR spectrum of propargyl alcohol, originally one of the starting substances, is shown in Figure 2 below.

![Fig. 2. Propargyl alcohol IR spectrum.](imageURL)

In the IR–spectrum of propargyl alcohol, in the area of 3290 cm⁻¹, a strong deformation oscillation belonging to the –OH group was manifested. Also in the spectrum there are weak valence oscillations of the –CH₂ group in the 2927–2870 cm⁻¹ field range, and deformation oscillations of the same group were observed in the 1448–1410 cm⁻¹ field.

The deformation absorption lines in area 2119 cm⁻¹ belong to the –C≡C– group. In addition, moderate intensity deformation oscillations of the HO–CH₂– group can be observed in the 914–638 cm⁻¹ field in the spectrum.

Figure 3 below shows the IR spectrum of the 2,3,3–triiode propene–2–ol–1 obtained.
The IR–spectrum of the 2,3,3–triode propene–2–ol–1 sample, in the area of 3190 cm\(^{-1}\), showed a strong deformation oscillation belonging to the –CH\(_2\)–C=C– group. There are also weak valence oscillations of the –CH\(_2\) group in the 2918 cm\(^{-1}\) field in the spectrum, and deformation oscillations of the same group were observed in the 1443 cm\(^{-1}\) field. The deformation absorption lines in the 1620 cm\(^{-1}\) area, however, belong to the –C=C– group.

In addition, in the spectrum, valence asymmetric oscillations of the 1543 cm\(^{-1}\) area – HC=CH\(_2\) group and deformation oscillations of the 779–544 cm\(^{-1}\) area of moderate intensity can be observed in the field.

In addition, the structure of the substance was also studied by the method of NMR–spectroscopic analysis. The following Figures 3–4 show the NMR–spectra of 2,3,3–triode propene–2–ol–1.

![Fig. 3. IR spectrum of 2,3,3–triode propene–2–ol–1.](image1)

The IR–spectrum of the 2,3,3–triode propene–2–ol–1 sample, in the area of 3190 cm\(^{-1}\), showed a strong deformation oscillation belonging to the –CH\(_2\)–C=C– group. There are also weak valence oscillations of the –CH\(_2\) group in the 2918 cm\(^{-1}\) field in the spectrum, and deformation oscillations of the same group were observed in the 1443 cm\(^{-1}\) field. The deformation absorption lines in the 1620 cm\(^{-1}\) area, however, belong to the –C=C– group.

In addition, in the spectrum, valence asymmetric oscillations of the 1543 cm\(^{-1}\) area – HC=CH\(_2\) group and deformation oscillations of the 779–544 cm\(^{-1}\) area of moderate intensity can be observed in the field.

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![Fig. 4. 1H NMR–spectrum of 2,3,3–triode propene–2–ol–1.](image2)
When analyzing the \(^1\)H NMR–spectrum of 2,3,3–triiodopropene–2–ol–1, proton signals (600 MHz, chloroform–d:CCl₄, \(\delta\), m.u., J/Gs) –hydrogen atoms in the CH₂– group 4.29–4.28 m.u., the hydrogen atoms belonging to the –OH group in the compound are 3.34–3.32 m.u. at intervals, the corresponding signals were detected. In addition the compound contains a double bond–bonded 2–position carbon of 117.30 m.u., The carbon belonging to the –CH₂– group is 70.43 m.u., While carbon in position 3 is 21.28 m.u. showed the appropriate signals in the sari. The obtained Spectra confirm the structure of the synthesized substance 2,3,3–triiodopropene–2–ol–1.

Fig. 5. \(^{13}\)C NMR–spectrum of 2,3,3–triiodopropene–2–ol–1.

4 Conclusion

On the basis of propargyl alcohol, the influence of various factors on the process of synthesis of 2,3,3–triiodopropene–2–ol–1 was studied and the optimal conditions were determined. Among the alkalis (LiOH, NaOH, KOH, RbOH) tested as catalysts, the product's yield was found to be high under the influence of NaOH. The effect of catalysts was studied in various concentrations, and the most effective concentration among them was found to be a 15% solution of NaOH. The reaction duration was studied between 1–5 hours and the temperature between 10–60°C, and the reaction duration was found to be 3 hours, with a high product yield when the temperature was carried out at 20°C. The structure of the synthesized substance was studied and the structure was confirmed by NMR and IQ–spectroscopic methods.

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