Nature of Invention: Chemical molecule and synthesis route

Applicant: BCG

Inventor 1: Amit Kumar Jana

Inventor 2: Priyansh Singh

Chemical Formula: C11H24O3

Chemical Name: Ethylhexylglycerine

Chemical synthesis routes:

Synthesis at lab scale :

Raw materials and chemicals

- Glycerin
- 2-ethylhexanol
- Sodium hydroxide (NaOH)
- Ethyl acetate
- Epichlorohydrin
- Catalyst – Boron trifluoride (BF3)
- Solvent – Dimethylsulfoxide (DMSO)

Reaction steps :

1. **Preparation of 2-ethylhexylglycidyl ether:**
   - Stir 2-ethylhexanol, sodium hydroxide (NaOH) and a phase transfer catalyst.
   - Add epichlorohydrin dropwise.
   - Carry out the reaction at 20° to 60° C for 3 to 20 hours to synthesize 2-ethylhexylglycidyl ether.
   - Filter the reaction product to obtain a filtrate containing 2-ethylhexyl glycidyl ether, then wash with water.
   - Distil the washed filtrate to obtain 2-ethylhexylglycidyl ether.
2. **Stir 2-ethylhexylglycidyl ether, water, organic solvent and catalyst:**
   - Stir 2-ethylhexyl glycidyl ether, water, an organic solvent and a catalyst. It is preferably carried out for 20 to 40 minutes under a nitrogen atmosphere.
   - The addition ratio of the 2-ethylhexyl glycidyl ether, water and the organic solvent is preferably 60-160 parts by weight of water and 10-33 parts by weight of the organic solvent, relative to 1 part by weight of 2-ethylhexyl glycidyl ether. When the above conditions are satisfied, the amount of ethylhexylglycerin produced can be increased.
   - It is preferable that the catalyst is Boron trifluoride (BF3).
   - Organic solvent is preferably Dimethyl sulfoxide (DMSO).
3. **After stirring, carry out the reaction at 80 to 120° C for 15 to 25 hours to synthesize ethylhexylglycerin:**

Below 80°C, the amount of ethylhexylglycerin produced is very low, and above 120°C, it is not economical.

4. **After the synthesis, add ethyl acetate:**
   - Adding ethyl acetate after the above synthesis removes excess water, the organic solvent and the residual catalyst after the synthesis, and extracts the compound.
   - Add ethyl acetate and let the mixture stay at room temperature for 20 to 40 minutes. Water and the catalyst get separated into the lower layer and ethylhexylglycerin is separated into the upper layer (organic layer).
5. **Remove the separated water layer, wash the organic layer:**
   - It is done for obtaining high purity ethylhexylglycerin.
   - It is preferable to add distilled water as wash water to the organic layer and then perform the treatment until the pH of the wash water becomes 5.5 to 7.0.
   - A dehydration process may be further included to remove moisture remaining in the organic layer. For example, 3 to 10% by weight of Na2SO4, followed by stirring and filtration for dehydration.
6. **Distil the washed organic layer to obtain ethylhexylglycerin:**
   - It is done for obtaining high purity ethyl hexyl glycerin.
   - Distil the washed organic phase at 25 to 35 torr and 70 to 90° C and then subject it to secondary distillation at 0.1 to 1 torr and 140 to 160° C. The primary distillation is to distil the remaining ethyl acetate, and the secondary distillation is to distil ethylhexylglycerin. Thus, ethylhexylglycerin obtained by the second distillation has a purity of 99.56 to 99.63%.

Alternative route: Ethylhexylglycerin can also be synthesized by using the starting material as 2-ethylhexanol. It is first mixed with the catalyst and epichlorohydrin is added drop wise under cooling. This results in the formation of 2-Ethylhexyl glycidyl ether. The second step is a ring opening step where, the obtained product is treated with water. As a result, the layers get separated to give the crude product. It is then purified to obtain the final qualified product which is then characterized. The scheme is shown below in the **fig 1.**

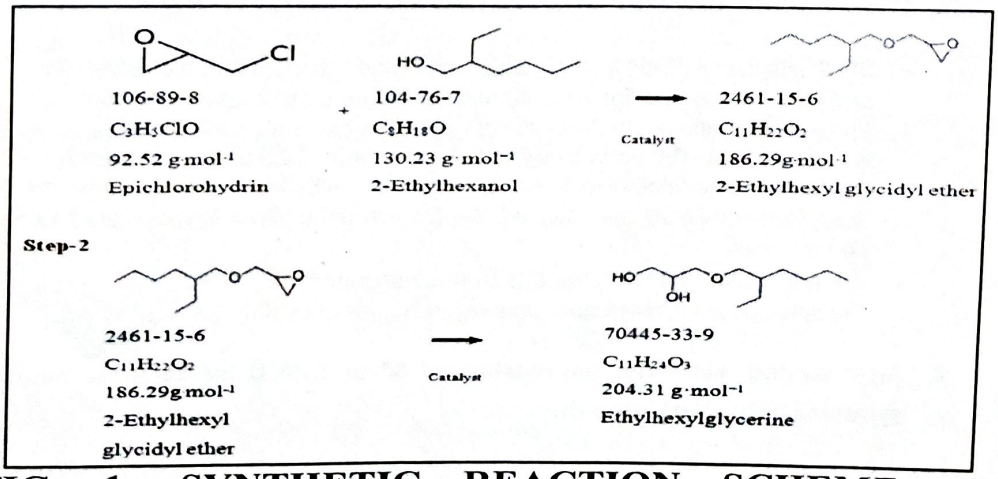

FIG. 1: SYNTHETIC REACTION SCHEME OF ETHYLHEXYLGLYCERIN

References: Example Patent : KR101528751B1 (Title : Method for producing ethylhexylglycerin)

References: research article ::IJPSR (2018), Volume 9, Issue 4, E-ISSN: 0975-8232; P-ISSN: 2320-5148

List the contributions of each author:

Amit Kumar Jana :

- Carried out the literature search, found out the reaction steps and product yield.
- Designed the chemical synthesis route.
- Found necessary separation steps to achieve desired product purity.

Priyansh Singh

- Designed the alternative synthesis route to obtain the compound in pure form.
- Worked upon the diagram and chemical structures of the reactants , intermediates ,products in the synthesis route.

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Name	Roll No	Signature
Abhinaba Mukherjee	210028	
Amit Kumar Jana	210122	
Priyansh Singh	210775	