STATEMENT

I hereby declare that the matter presented in this dissertation entitled "Synthesis of Novel Polysulfone monomer, Bis(2-hydroxyethylsulfone),2-dimehylpropane" is based on the results and investigations carried out by me in the School of Chemical Sciences, Goa University under the supervision of Prof. V.S. Nadkarni and the same has not been submitted elsewhere for the award of a degree/diploma.

(Elaine Coelho)

CERTIFICATE

This is to certify that the dissertation entitled "Synthesis of Novel Polysulfone monomer Bis(2-hydroxyehylsulfone)2,2-dimethylpropane" is a bonafied work carried out by Miss Elaine Coelho, under my supervision in partial fulfilment of the required the award of the degree of Master if Science in Organic Chemistry at the School of Chemical Sciences, Goa University.

Dr. V.S Nadkarni

Dean, School of Chemical Sciences

Project Supervisor

CERTIFICATE FROM DEAN

This is to certify that the dissertation entitled "Synthesis of Novel Polysulfone monomer Bis(2-hydroxyethylsulfone)2,2-dimethylpropane" is a bonafied work carried out by Miss Elaine Coelho for the award of the degree of Master of Science in Organic chemistry at the School of Chemical Sciences, Goa University.

Dr. V.S Nadkarni

Dean, School of Chemical Sciences

ACKNOWLEDGEMENT

The work carried out in this Project would not be possible without the co-operation, hardwork and determination of the student as well as the teachers. I am extremely fortunate to have got the required assistance I needed throughout my project period.

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Finally I am thankful to my **parents and my friends** for supporting and encouraging me and the **Almighty God** for blessing me.

CONTENTS

CHAPTER ONE: INTRODUCTION

Since the beginning of the onset of civilization and birth of industrialisation, the replacement of primitive and natural materials has played a key role in modernization and easing of lifestyle. Polymers in this respect have made life much easier and have always been a fascination to man and revolutionised the time and cost of production, materials we use and in short impacted our daily lives and the environment we live in. It is justified to say that polymers have been the backbone units from the biomolecules linked to eachother within our body right up to the macromolecular scale like the plastics or construction materials we use, in addition to also forming the basis of the four major Chemical industries, namely- Plastics, fibres, Elastomers, Paints and Varnishes.

Polymers are large molecules having a molecular mass around 10^3 to 10^7 u and are built up by a repetition of small, simple chemical units (Monomers) tgat are held together by Covalent bonds. The process by which these molecules are linked to eachother and converted to polymers is called Polymerisation and can result in either linear chains or inter-linked or 3-D networks.

Owing to the vast applications of Polymers in fields like avionics, packaging, tissue engineering, implants, bio-sensors, drug delivery systems and so on, we wish to synthesis a novel monomer Bis(2-hydroxyethylsulfone)2,2-dimethylpropane that contains several sulfone groups that could be further polymerised into a 'Polysulfone polymer' and to study the potential properties and applications of the resultant molecules. The Novel thing about this monomer is that unlike already pre-existing sulfone polymers, this monomer unit doesn't possess any aromatic ring within its structure and is completely aliphatic.

Classification of Polymers

There have been several different basis for the classification of Polymers the most common being the following :

A) Based on Origin :

1. Natural Polymers :

These are the polymers that are obtained naturally in nature from plant and animal sources. Common examples of natural Polymers include Natural rubber, starch, cellulose and biomolecules like proteins.

2. Synthetic Polymers:

Man-made polymers that are either partially or completely derived from synthesis artificially in laboratories and are prepared for a certain purpose mostly fall under the category of Synthetic Polymers. These include commonly used polmers like Polyethylene, polystyrene, Teflon, PVC and so on.

B) Classification based on Mechanical Strength :

1. Thermoplastics:

These type of Polymers can be moulded into a variety of shapes on changing the temperature and pressure, with little changes in their properties. This is possible die to absence of cross linking between the individual chains. Common examples include Polystyrene and Teflon.

2. Thermosetting Plastics:

On application of temperature and pressure these Polymers can be moulded only onc and undergo permanent change in their structure. Therefore they are not suitable for reusing. There is extensive cross linking and thus these Polymers are highly branched and often rigid as seen in examples like Melamine Formaldehyde resins and Bakelite.

3. Elastomer:

Elastomers can be easily stretched on the application of small amounts of stress and immediately regains it's original shape when at ease. Thus they have high reversible extensibility. The amount of elasticity is directly proportional to the temperature and is credited to the presence of weak forces holding the polymer together. The most common example is Natural Rubber.

4. Fibres:

These type of Polymers show low elasticity and high tensile strength and are crystallizing nature. They show permanent effect due to deformation due to the presence of stronger intermolecular attraction between the monomer molecules. Example is Nylon-6,6

C) Classification based on Structure

1. Linear Polymers:

In linear polymers the monomer units are arranged end to end to form long linear chains. These Polymers are mostly crystalline, have high density, high tensile strength and melt at high temperatures.

2. Branched Polymers :

Polymers maybe arranged linearly but the peculiarity of branched Polymers are the presence of side chains of varying lengths. These Polymers melt relatively lower than linear polymers and possess lower tensile strength as well.

3. Cross-linked Polymers:

In these Polymers cross linking takes place between two strands of monomer units to form a three dimensional structure. These Polymers are hard, rigid and difficult to melt even at high temperatures due to the extensive cross linking.

D) Classification based on Methods of Production:

1. Addition Polymers:

These Polymers are formed with the repeated addition of monomer units, retaining their identity without the elimination of any by-product molecule. Examples of such polymers include PVC, polyethylene, polypropylene, polystyrene.

2. Condensation Polymers:

These Polymers are a result of a condensation process between two monomer units and the elimination of a small by-product molecule mostly water, ammonia or HCl. The most common example of this polymers are polyesters and polyamides.

Polymerisation Techniques :

A) Bulk Polymerisation: This technique employs the use of moulds of required shape and size into which the monomer is taken and heated or exposed to radiation to initiate Polymerisation. This technique gives high purity and insoluble initiator. The use of chain Transfer reagents helps in the control of the molecular weight of the polymer. Bulk Polymerisation is an exothermic process, therefore more and more heat is generated that increases the viscosity, which makes mixing difficult and a broad molecular weight distribution is obtained. Termination is also equally difficult in the case of Bulk Polymerisation. PVC, condensation of polystyrene and polyethylene methacrylate are commonly prepared by this technique.

B) Solution Polycondensation: The shortcomings of the Bulk Polymerisation Technique such as problem of heat dissipation is overcome in Solution Polycondensation where in a suitable solvent dissolves thr monomer, initiator as well as the polymer. This ensures homogeneity of the system. The mixture is kept at the Polymerisation temperature and constantly agitated and the droplets are prevented to coalesce by adding a small amount of stabilizers. This technique is widely used for the production of Polymers like Polyvinyl Alcohol, Polyvinyl Chloride and polystyrene.

C) Suspension Polymerisation: In this technique, the monomer droplets are stabilised by the addition of surfactants or protective colloids. This technique can only be used in the case of insoluble monomers. The size of monomer droplets can vary from 50-20 um in diameter. The initiators used are monomer soluble and thus the polymer produced is a heterogeneous system. Since each monomer droplets acts as a separate Bulk Polymerisation nucleus, this ensures excellent heat transfer to water. The purity of such polymers are however low due to trapped impurities. PVC, Styreme-acrylonitrile are prepared by this method of Polymerisation.

D) Emulsion Polymerisation: This is similar to Suspension Polymerisation In the fact that the Emulsion of monomer in water is stabilised by a surfactant. But here, the monomer is dispersed in the aqueous phase as a uniform Emulsion and not as discrete droplets. The droplets of monomer are emulsified by soap which forms micelle, in which the polar hydrophilic ends orient outwards (towards water) and the hydrocarbon hydrophobic ends orient themselves inwards. Such a system contains unsolublised monomer droplets too and they enter the micelles as Polymerisation continues. The Polymerisation starts at the surface and proceeds inwards. As monomer inside the micelles is consumed, monomer droplets diffuse into the micelle. The chain ends when another initiator molecule enters inside the micelle and arrests chain growth. After sufficient growth of the micelle, it bursts into a milky white suspension called 'latex'. This technique has the advantage of high rates of Polymerisation and high molecular weight product can be obtained. It is widely used in the case of monomers like vinyl chloride, vinyl acetate and acrylates.

E) Melt Polymerisation: This technique can be used in the case of Monomers that do not decompose around their melting point. Inert atmosphere and reduced pressure is necessary while carrying out this Polymerisation. The pure molten Monomers are polymerised in the absence of added solvents and is advantageous because The molten polymer formed is required to be discharged hot from the reactor and can be directly passed for spinning, casting, extrusion or spun into fibres. This makes it an environmentally friendly process than other industrial Polymerisation Techniques. Polyethylene terephthalate and Nylon-6,6 are prepared by this technique.

Polysulfones Polymers and their Applications

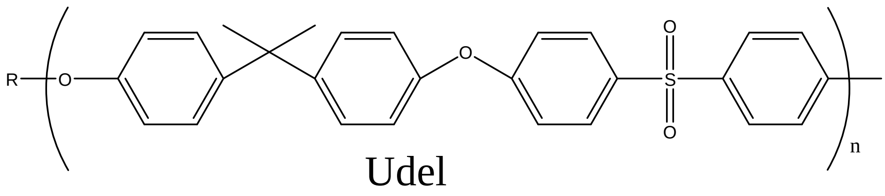
Polysulfones are a type of thermoplastic polymers that contains recurring sulfone groups(-SO_2) in the main chain. When we talk about polysulfones, we mostly only refer to aromatic polysulfones in the technical context. The aromatic rings include p-phenylene groups that may be joined end to end at para position through Sulfone groups (this gives us the simplest polymer poly(p-phenylene)sulfone or may include alternating ether linkages as well (polyarylethersulfones)

Sulfone groups are highly polar and contain sulfur in its highest oxidation state and hence are resistant to further oxidation. Polymers that contains both Sulfone groups and p-phenylene groups are expected to have a high glass transition temperature. For example, poly(p-phenylene)sulfone melts at 520°C, but with decomposition. (Not a good thermoplastic) It is therefore necessary to make these polymers less stiff and this is achieved by incorporating ether linkages within the polymer.

Polysulfones were first introduced by Union Carbide in 1965. There are five major types of Polysulfones made by three companies. Union Carbide made polysulfones (Udel and Radel) in 1965 and 1976 respectively, ICI made polyethersulfones (Victrex and Polyether sulfone 720P) in 1972, Minnesota Mining and Manufacturing (3M Corp) made polyarylethersulfones (Astrel) in 1967.

Udel:

Udel is a thermoplastic resin possessing one of the highest service temperatures. (approximately 170° C) It is highly resistant to mineral acids, salts, alkali, oils, detergents and electrolytes. But is affected by organic solvents and other aromatic hydrocarbons.Udel properties can be summarized as having high thermal stability, good toughness, stiffness and tensile strength, it is environmentally stress resistant as well as flame resistant and transparent.

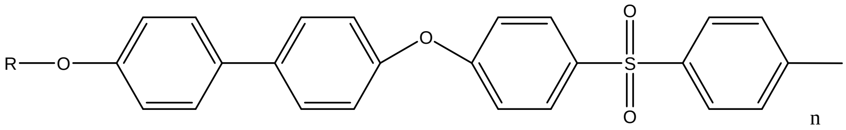


Radel:

Radel is a high performance,yet lightweight polysulfone thermoplastic.Radel was first synthesised in 1976 from the reaction of a bisphenol and bis(p-chlorophenyl)sulfone and exhibted a greater chemical and solvent resistance than Udel. It has higher resistance to steam and soldering temperatures than any other thermoplastic. Radel can withstand high temperatures during steam distillation or autoclaving with little to no dimensional loss ot instability, thus making it a suitable candidate for medical devices. It is anideal material for all types of medical instrumentation. Moreover Radel is transparent and available in clear and custom colours. It can uptake any colour deppending on the purpose and is used in dental and surgical tools and instruments. It is also used in other medicsl applications like insulin pens, blood glucometer tubings, pharmaceutical equipment, fluid handling couplings and fittings.

Radel also has a high dielectric strength that makes it an excellent choice for electronic equipment and devices. It has excellent resistance to heat and environmental stress, making it useful in electronic assembly, machining and finishing.

However use of Radel is not considered appropriate for food contact applications and its properties are known to degrade when exposed continuously to sunlight.

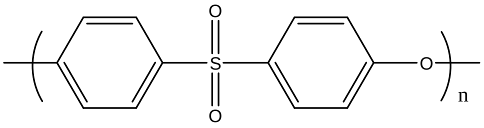


Radel

Victrex 450 P

Victrex 450 P has been one of the first polyethersulfones to be synthesised. Till date, owing to its hard, stiff and light weight properties, it has found greatest applications in the automotive industry. Victrex thermoplastic polymers retain their original stiffness and tensile strength up to high temperatures (343°C) allowing them to perform under aggressive environments. Instead f using metals like aluminium alloys, use of Victrex provides by far the lowest weight per volume ratio. Victrex is also usEd to form tribological components due to its resistance to wear under pressure.

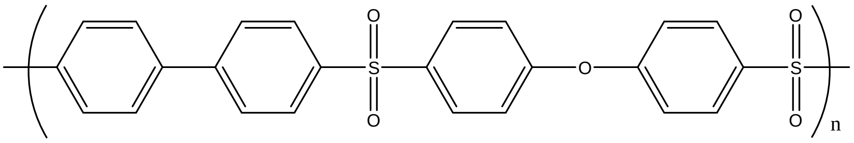
Parts made of Victrex are comparatively more economical to produce, reduces costs, weight and use of metals, recurrent maintainence, enhanced fuel consumption, Lower CO_2 emissions, reduces noise, increases the longetiviety of the instruments or machinery and being a thermoplastic is recyclable.



Victrex

Astrel

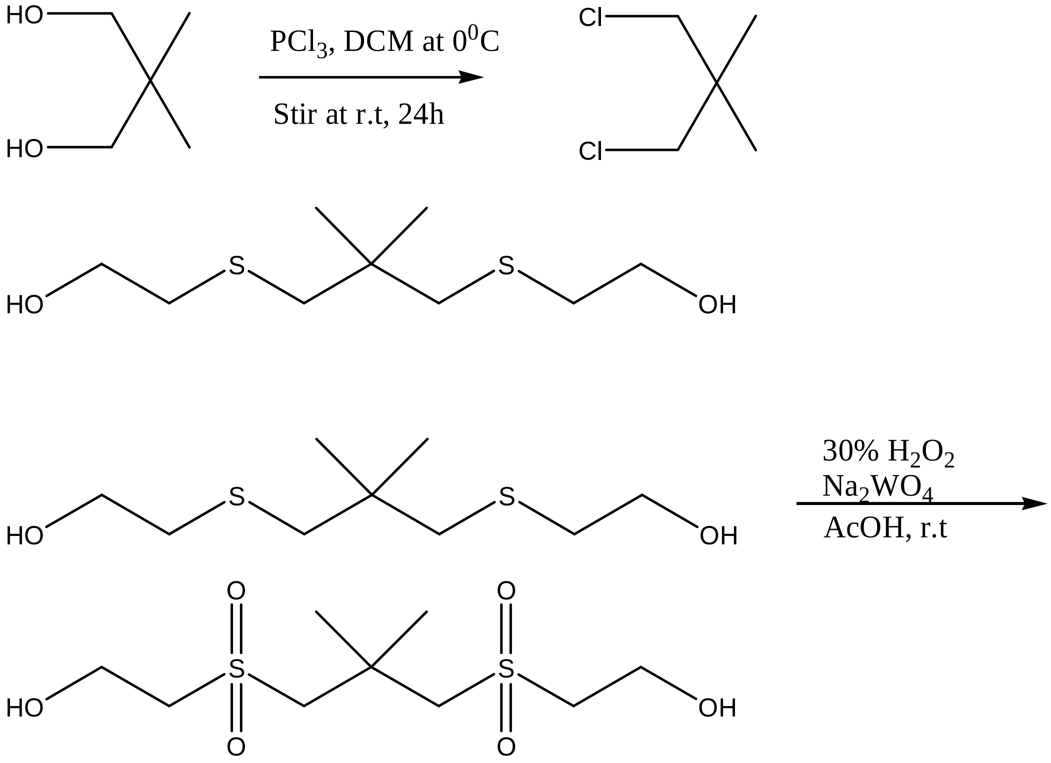
Astrel 360 was the first high performance thermoplastic to be synthesised by the 3M Corp. It is known for its stiffness and durability and has been widely used in the manufacture of pipes to transport water,steam due to its high tolerance to high temperatures without suffering any deformation in particular.



Astral

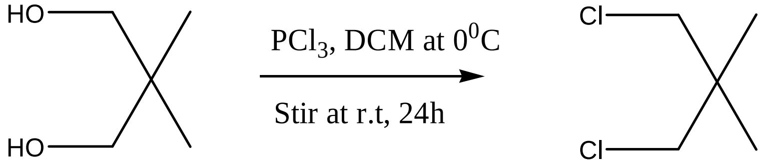
CHAPTER TWO : LITERATURE STUDIES

General Synthetic Route of target monomer Bis(2-hydroxyethylsulfone)2,2-dimethylpropane:



Stepwise over view of the reaction :

1. **First step: Chlorination of Neopentyl Glycol**



2,2-Dimethyl-1,3-propandiol **1,3-dichloro-2,2-dimethylpropane**

In the first reaction Step 2,2-dimethyl-1,3-propandiol (Neopentyl glycol) is chlorinated with the help of 2 reagents, either Thionyl chloride or PCl_3.

1. In a 250 ml round bottom flask, 3g of neopentyl glycol (22.03 mmol) dissolved in dichloromethane was taken and kept at 0°C in an ice bath.
2. 20ml of PCl3 (excess) was slowly added in dropwise and the solution was stirred at rooms temperature for 24 hours.
3. The solvent was then removed by distillation to yield halogenated white precipitate of 1,3-dichloro-2,2-dimethylpropane.

The above reaction can also be carried out using Thionyl chloride adopting Triphenylphosphine oxide as catalyst, the only difference being a increase in temperature upto 65-90°C, and Stirring for 2 hours.

Reaction required to be added

The use of Triphenylphosphine oxide (TPPO) in chlorination reactions is in accordance with a new Phosphine-oxide catalysed chlorination reaction of Alcohols under Appel conditions. The reaction is effective for acyclic primary and secondary alcohols and generated 2 gases as by-products, SO_2 and HCl.

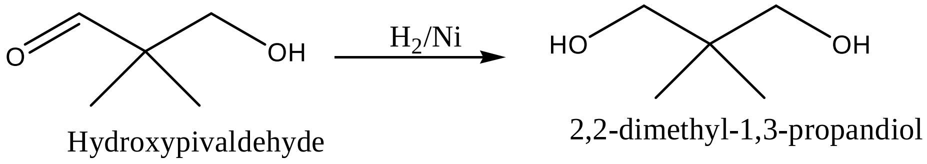
The driving force for chlorination under Appel conditions using chlorophosphonium to convert alcohols to chlorides is the formation of strong P=O in the Phosphine-oxide. Harsh conditions are required for the reduction of Phosphine-oxide to Phosphine from which chlorophosphonium salts are conventionally prepared. TPPO is first transformed into the required Chlorophosphonium reagent on reaction with $SOCl_2$. It has been seen in literature that chlorotriphenyl phosphonium chloride is rapidly generated in-situ from Triphenylphosphine oxide and this reagent is effective at chlorinating primary alcohols like decanol.

Neopentyl glycol

Synthesis

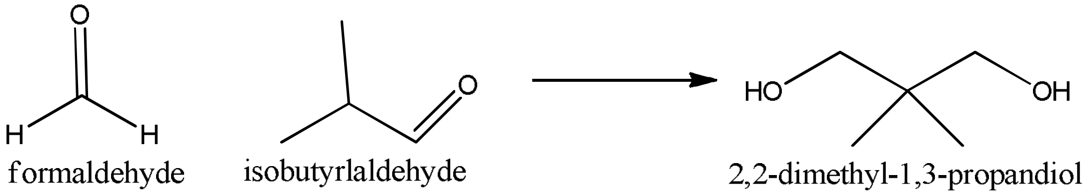
(A). By catalytic hydrogenation of hydroxyl aldehyde ethanol

Nanonickel phosphate is filled in a fixed bed reactor which was treated in hydrogen gas flow at 200°C for 2 hours, simultaneously feeding hydroxypivaldehyde ethanol solution and reacting it at 80-130°C and 0.5-3 MPa to obtain Neopentyl glycol.

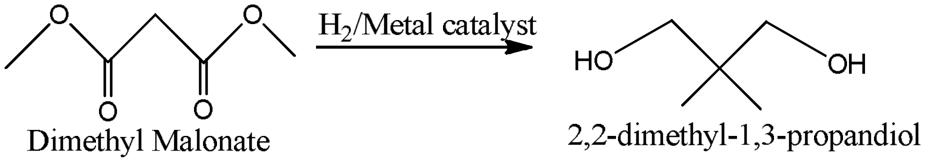


(B) By Aldol Condensation of formaldehyde and isobutrylaldehyde

37% aqueous formaldehyde solution and 99% isobutrylaldehyde was taken at a mass ratio of 1.25:1 and the pH was adjusted to 6-8.5 and Aldol Condensation reaction was carried out at 60-80°C for 30 minutes to 2 hours, to obtain an intermediate which on catalytic hydrogenation yields crude Neopentyl glycol.



(C) By Catalytic hydrogenation of dimethyl malonate



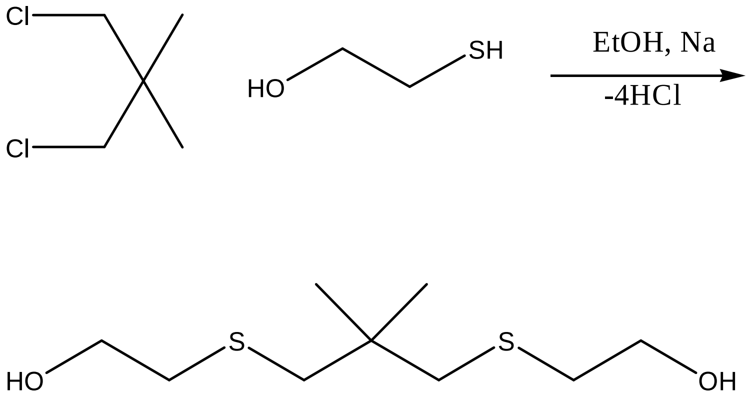
To a mixture of silica bound metal catalyst (Ru:Sn:Pt) in the ration of 1:1:1.8 wt% (25g) , dimethyl malonate (5g) and 100g water was added in a high pressure reactor and supplied with hydrogen, the resultant mixture was stirred (210°C, Hydrogen at 100 Bar for 3 hours) and cooled at room temperature and after removal of water, Neopentyl glycol was obtained.

Commercial Applications of Neopentyl glycol

Neopentyl glycol is an important Commercial organic compound. It is a colourless to pale white, hygroscopic crystalline organic compound that is used in the synthesis of paints, polyesters, lubricants and plasticisers.

Its industrial uses include dyes, functional fluids in a closed system, as intermediates, additives in lubricants and coatings and processing aids specific to petroleum production. Consumer uses include adhesives and sealants and manufacturing paper products.

2. **Second Step** :



In the second step of the reaction 1,3-dichloromethyl-2,2-dimethylpropane is further reacted with 2 molecules of 2-mercaptoethanol leading to the formation of the desired product. The above reaction was carried out using the following procedure.

1. To 200 parts Ethanol, 4.6 parts of metallic Sodium was added along with 16 parts of 2-mercaptoethanol and 19.4 parts 1,3-dichloromethyl-2,2-dimethylpropane.
2. The reaction mixture was heated under reflux
3. The deposited NaCl was removed by adding a non polar solvent and filtering the solution and solvent ethanol was removed by distillation to give us product Bis(2-hydroxyethylthiomethyl) propane.

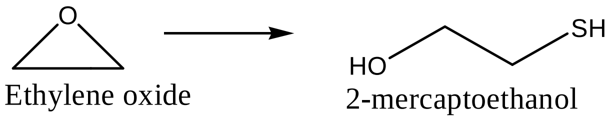
Another alternative for the preparation of bis(2-hydroxyethylthiomethyl)propane would be by the attack of Sulphur atoms lone pairs of 2,2-dimethyl-1,3-propandithiol directly on ethylene oxide which would result in opening. This route cuts down our reaction procedure by 1 step and we would easily get our desired monomer by carrying out the third oxidation step on it. However the only drawback suffered would be the preparation and instability of ethylene oxide and careful control of reaction conditions.

2-Mercaptoethanol

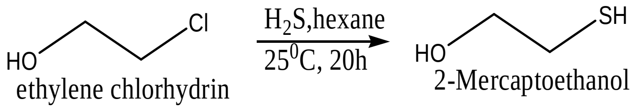
Synthesis

(A) By the reaction of ethylene oxide and hydrogen sulphide

1. In a 1L autoclave, 200g hexane and 1g of ion exchange resin was added and the mixture stirred at a temperature of 25°C.
2. To this hydrogen sulphide and 56g of ethylene oxide was added dropwise over a period of 2 hours.
3. The mixture was allowed to stand for 20 hours at 25°C. Nitrogen was then bubbled into the autoclave to remove hydrogen sulphide gas.
4. The reaction mixture was filtered and a 2 phase liquid system was obtained as filtrate. The lower layer was 2-mercaptoethanol and the upper layer is organic layer of hexane. Results can be analysed by gas chromatography.



(B) A similar reaction can be carried out by using ethylene chlorhydrin instead of ethylene oxide along with Sodium or potassium hydrogen sulphide.

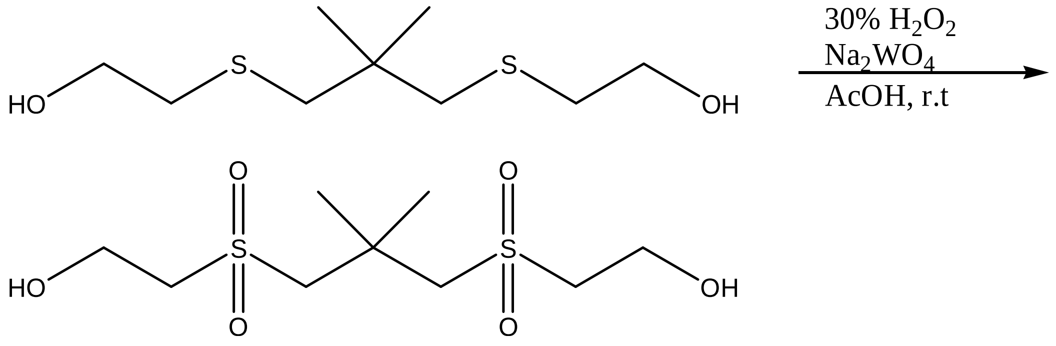


Commercial Applications of 2-mercaptoethanol

Beta mercaptoethanol is a organic solvent that is widely used in organic synthesis as well a biochemical reagent in various assays. It can act as a solvent as well as intermediate for producing dye stuffs, pharmaceuticals, rubber chemicals, floatation agents, insecticides, plasticisers, water soluble reducing agent, biochemical reagent, PVC stabiliser, as agricultural chemical and as textile auxiliary.

It is also employed as a biochemical research tool, corrosion inhibitor and anti-scaling agent and water treatment products.

3. **Third Step**



The oxidation of sulphur containing polymers into their respective sulphoxides and Sulphone derivatives is an effective way to increase the polarity of the resulting macromolecule and bestow them with superior electrical, mechanical and thermal properties.

In the Third and final step of the reaction, the sulphide groups of bis(2-hydroxyethylthiomethyl) propane are oxidised to sulfone groups using hydrogen peroxide. The solvent for the reaction is chosen based on the desired product, AcOH preferably leads to the formation of sulphoxides whereas CF_3COOH leads to formation of sulfones. The reaction was carried out using the following procedure :

1. In a 100mL Round bottom flask, 2mmol of our sulphide solution was taken in a 1:1 mixture of methanol and Dichloromethane.(10mL)
2. 30% of hydrogen peroxide solution (3-8 equivalents) was added along with Na_2WO_4 as catalyst.
3. The reaction mixture was stirred magnetically at room temperature. The progress of the reaction was monitored using TLC (Or GC as well)
4. After completion, the reaction mixture was filtered off and rinsed twice with 25-30ml of dichloromethane. Excess solvent was removed under reduced pressure.

Various reagents have been used for oxidising Sulphides to Sulfones which include peracids, $NaIO_4$, $KMnO_4$, CrO_3, HNO_3. However most of them aren't atom efficient, concerns high cost and may produce by-products that are not environmentally favourable. The most commonly used oxidising agent used is meta-chloroperbenzoic acid. (m-CPBA) But this reagent suffers certain drawbacks such as risk of over oxidation (risk being especially high due to presence of 2 hydroxyl groups that would be oxidised to aldehydes), partial solubility in solvents like DCM, strict temperature control and the difficulty to separate the by-product that is m-chlorobenzoic acid from the sulfone, which makes it an unsuitable reagent in this case.

In contrast, oxidation of sulphides using H_2O_2, is the most attractive because it is cheap, environmentally benign, safe in storage and operation, readily available and easy to handle with an efficient oxygen content. In addition to Na_2WO_4, which is the most easily available catalyst used along with 30%H_2O_2, various other catalyst and combinations can be used. Catalysts have been imobilised on silica and other solid supports that serve the purpose of simple catalytic separation, long catalytic life, high reusability and thermal stability. Different supports include, organic polymers like polystyrene, resins, mesoporous silica (MCM-41). For example, a prepared combination of a Cu(II) Schiff base complex was imobilised on amino-modified MCM-41, to give us the catalyst (MCM-41-pr-NH_2-CuL) where L= salicylidineiminoethyliminopentane-2-one)

The above catalysts can also be coupled with the use of ultrasonic waves. The essential role of ultrasonic waves in the synthesising of organic compounds is well known. It has been demonstrated that the conversion and selectivity of the wanted product can be improved under ultrasonic irradiation. The main effects of ultrasonic irradiation in the liquid such as a local enhancement in temperature and a local high pressure are due to the 'cavitation' phenomenon. These effects would lead to a particle size decrease as well as area-enhancement and accelerated motion of the suspended particles leading to a better transfer of solid-liquid mass. Therefore the use of ultrasonic waves can accelerate the reaction speed.

Procedure: (using Ultrasonic irradiation)

In a test tube, sulphide solution (1mmol), 100 mg catalyst (0.012mmol) and 30% H_2O_2 (2mmol) were mixed in MeOH. Then the reaction mixture was exposed to ultrasonic irradiation for 30 minutes to an hour. Checking of the reaction progress was accomplished by using n-hexane: ethyl acetate (Various ratios). The solid catalyst was filtered after the completion of the reaction and the product was extracted using ethyl acetate. (3×10mL) and refined on a silica gel plate. Finally the conversion and selectivity values of products were determined using GC.

Another reagent that is often used for the selective oxidation of sulphides to sulphones is oxone (Potassium peroxomonosulphate) ($K_5H_3O_{18}S_4$) Many procedures that employed oxone as an oxidising agent required the use of a catalyst as well, but the most simplest, easily available and clean option being the use of a base-oxone combination.

Procedure: To a well stirred mixture of sulphides (1mmol), a selected base catalyst (0.02mmol) in acetonitrile (2mL) was added to the solution of oxone (2mmol) in 4 mL of water. The reaction was monitored by TLC.

From all the organobasic catalysts that were studied including DBU, piperidine, diethylamine, DMAP, triethylamine, it was found that based upon parameters of cost, commercial availability, ease of handling and ease of removal from the reaction mixture during work-up, diethylamine was the best suited catalyst.

CHAPTER THREE : EXPERIMENTAL WORK

Preparation of Triphenylphosphine oxide (TPPO) :

A solution of 0.1468g of Triphenylphosphine and 20mL DCM was reacted to form ylide and treated with 6mL of 3% hydrogen peroxide and stirred for 3 hours. The aqueous layer was discarded and The solvent was evaporated from the organic phase to yield TPPO as a white solid.

Chlorination of Neopentyl Glycol to 1,3-dihloro-2,2-dimethylpropane.

- A mixture of 4g Neopentyl Glycol and 0.5898g of TPPO were placed in a flask and at 80°C thionyl chloride was added drop wise within 2 hours with constant stirring.
- The temperature was further raised to 90-100°C and the mixture was additionally stirred for 3 hours. After cooling to room temperature, water was added carefully followed by conc. solution of Saturated $NaHCO_3$ (approx 50 mL)
- The mixture was extracted thrice with DCM. The combined organic phases were washed with water, dried over Na_2SO_4 and solvent was removed by normal distillation to give product as a pale yellow liquid

Conversion of 1,3-dichloro-2,2-dimethylpropane to . Bis(2-hydroxyethylthiomethyl)propane

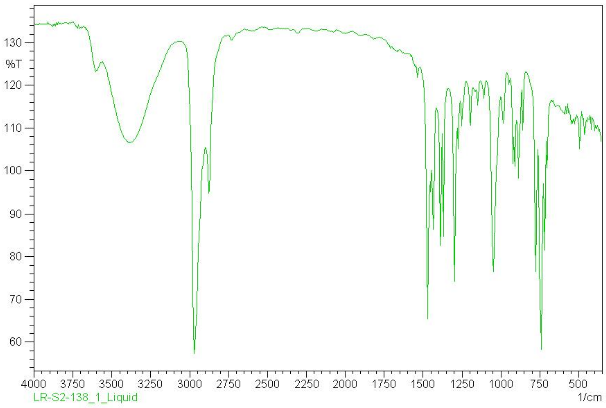
- A solution of 1.82 mL 1,3-dichloro-2,2-dimethylpropane in 200 mL methanol was added drop wise over 30 minutes stirring to a mixture of 4.5 mL mercaptoethanol and0.75mL of 11M NaOH.
- The reaction was stirred at 90°C for 3 hours. The solvent was evaporated under normal distillation and residue was poured in water and extracted with dimethyl ether.
- The combined ether extracts were washed with sat. NaCl and dried over Na_2SO_4. The filtrate was evaporated under vacuum.

RESULTS AND CONCLUSIONS:

1. The melting point of TPPO was found to be 156 °C in accordance to the reported range of 154-158°C

2. TLC carried out in 50% ethyl acetate showed spots for both reactants and products in all cases.

3. Test for halides showed Chloride present. However, test for alcohols also showed positive indicating presence of hydroxyl group.



IR Spectra of 1,3-dimethyl-2,2-dimethylpropane

IR spectra of Bis(2-hydroxyethylthiomethyl)propane

Due to COVID-19 pandemic, access to laboratory was stopped since March 2020 and further experiments could not be carried out.

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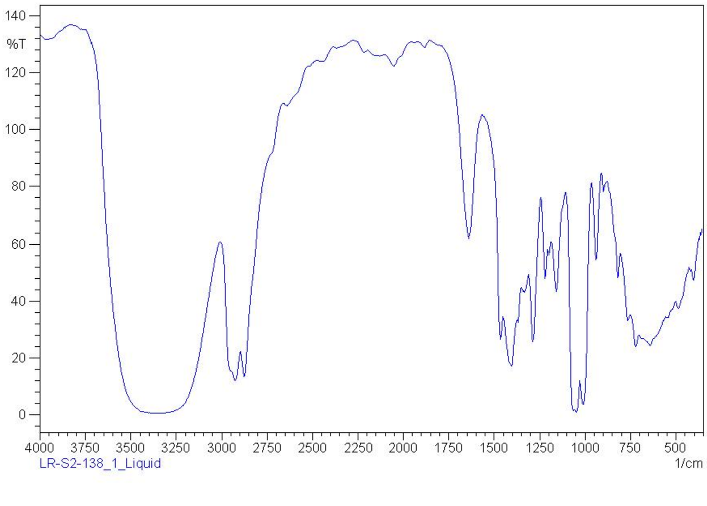

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