Significance and use of glycidol

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Please cited as: CHEMIK 2010, **64,** 5, xx-xx

Introduction

Glycidol is an epoxy compound with numerous applications. It may be obtained via chlorine or chlorine-free methods. Chloride methods generate a large amount of waste; they are very harmful to the environment. Chlorine-free methods, using allyl alcohol as an organic raw material, and whose oxidizers include peracids, organic hydroperoxides or hydrogen peroxide, also cause the necessity to manage a large amount of by-products. Currently, the Institute of Organic Chemistry Technology of the West Pomeranian University of Technology in Szczecin is conducting studies on creating a modern, coherent with the requirements of "green chemistry", technology of obtaining glycidol through oxidizing allyl alcohol with a 30% hydrogen peroxide on titanium-silicalite catalysts: TS-1, TS-2, Ti-Beta, Ti-MCM-41 and Ti-MCM-48. In this method, the by-product of the hydrogen peroxide conversion is water, and organic by-products may be managed easily. Epoxidation is carried out under mild conditions (temperatures up to 120°C and autogenic or atmospheric pressure). The titanium-silicalite catalyst can be recovered easily from the post-reaction mixture, regenerated and further turned back to the process.

Glycidol properties

Glycidol (2,3-epoxy-1-propanol) is a colorless and almost odorless liquid with a molar mass of 74.08 g/mol. It is the simplest chemical compound which includes an epoxy group in the particle, as well as a hydroxyl group in the α position with reference to the epoxy group [1].

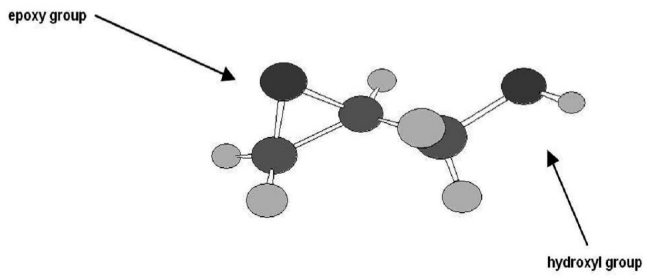


Fig. 1. The structure of glycidol molecule

Glycidol is characterized by the following physical properties:

- boiling point: 161-163°C (under a pressure of 760 mmHg), 67-69°C (20 mmHg), 57-59°C (10 mmHg)
- melting point 54°C
- density: $d^{20}_4 = 1{,}115$ g/dm^3
- refractive index: $n_D^{20} = 1{,}4311$
- viscosity in a temperature of 20°C = 4,00 cP

solubility: glycidol mixes in every proportion with water, lower alcohols, ketones, esters, dimethyl ether, benzene, toluene, styrene, chlorobenzene, methylene chloride, chloroform, acetonitrile; it dissolves poorly in aliphatic and cycloaliphatic hydrocarbons [2].

Glycidol reactions occurring with an epoxy ring opening

Glycidol belongs to a group of very reactive compounds. As a result of addition two particles of glycidol, glycerin-glycidol ether is created. The addition of further glycidol particles leads to the creation of a polyglycidol [3].

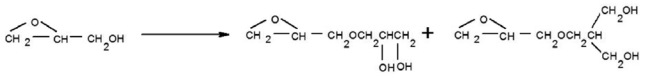


Fig. 2. Addition of glycidol molecules and polyglycidol formation [3]

These reactions occur even during the storage of glycidol. The decrease of the epoxy groups content caused by this reaction in a temperature of 25°C amounts to approx. 2% per month. By dissolving glycidol in such solvents as benzene, toluene, chlorohydrocarbons, it is possible to eliminate the occurrence of this reaction. Because of its bifunctional character, glycidol reacts as a 1,2-epoxide and like an alcohol and it is a material used for obtaining glycidol and glycerin derivatives. Thus, glycidol reactions may occur with the preservation of the epoxy ring or its opening.

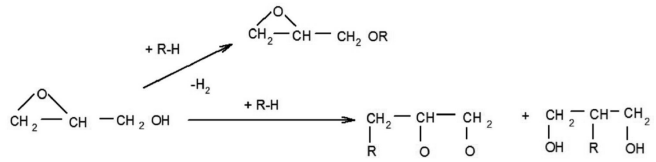
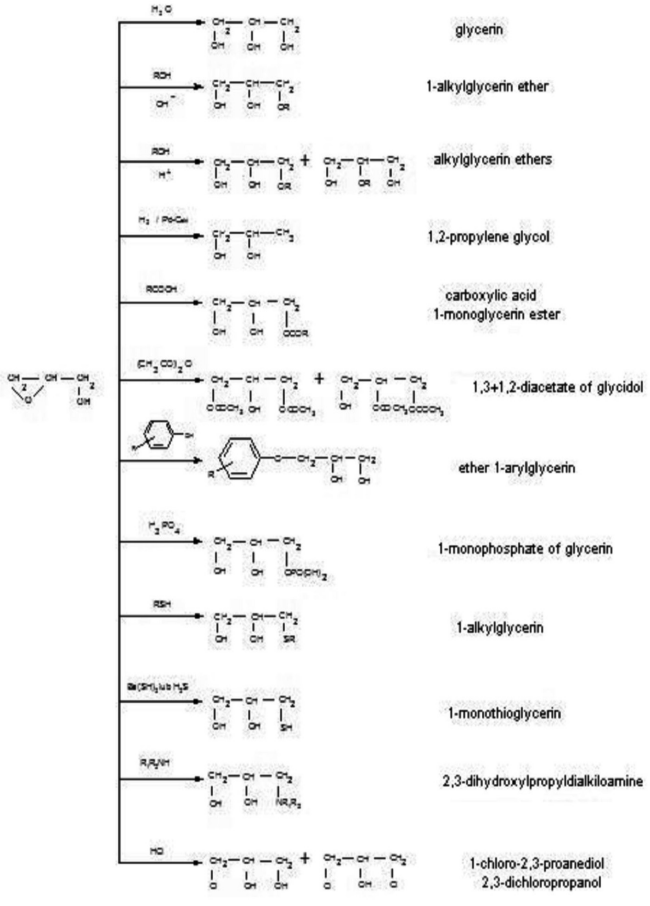


Fig. 3. Reactions of glycidol proceed with retaining or opening of epoxy ring [3]

A commonly known glycidol reaction occurring with the epoxy ring opening is hydrolysis to glycerin. In the presence of a surplus of water, it occurs with an almost 100% efficiency [4]. Glycidol reactions occurring with an epoxy ring opening have been presented in Scheme 1.



Scheme 1. Glycidol reactions occurring with an epoxy ring opening [3]

Reactions with nucleophiles, occurring with an oxirane ring opening, are catalyzed by acids and bases. In the presence of an acid catalyst, a mixture is created of 2,3 and 1,3 dihydroxypropylene compounds. In a base environment, mainly 2,3-dihydroxypropylene derivatives are created [3]. By using a base catalysis through the reaction of glycidol with alcohol, 1-alkylglycerin ether is created. In an acidic environment, a mixture of 1-alkyl- and 2-alkylglycerin ether is created. Primary C_1-C_{10} aliphatic alcohols create 1-alkylglycerin ethers, which are used in the varnish industry [5].

Depending on the temperature, it is possible to obtain glycerin and polyglycerins, or glycidol and polyglycidol [3].

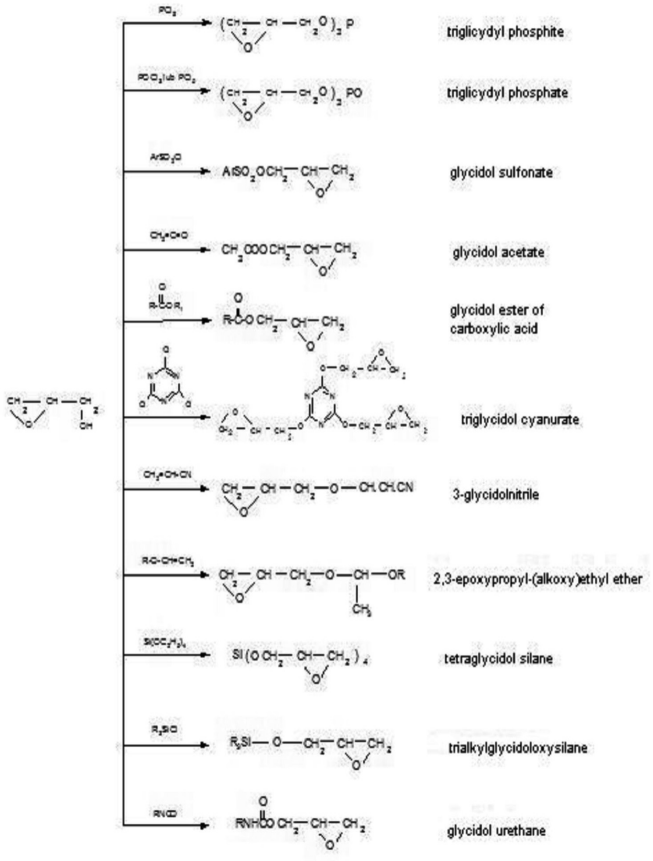
A catalytic hydrogenation of glycidol in the presence of palladium on an active carbon in an anhydrous ether as the solvent leads to the obtaining of a 1,2-propylene glycol. This glycol is used in the pharmaceutical, food, cosmetic, plastics and paper industry. In the reaction of glycidol with acid phosphate in a water solution, a 1-monophosphate of glycerol is obtained which is used for creating emulsifiers [3].

In the reaction of glycidol with fatty acids, glycerin monoesters are created which are used as emulsifiers, stabilizers and agents added to food.

During the reaction of an acetic acid anhydride with glycidol, a mixture of 1,2- and 1,4-diacetate of glycerin is obtained. The reaction of glycidol with alkylphenols, with the creation of 1-arylglycerin ethers, occurs easily in an alkaline environment. Compounds of such a structure constitute components of cough syrups. Alkylphenylpolyglycerin ether is used as a non-ionic surfactant. It is used for lowering the surface tension of aqueous solution liquors [6].

Glycidol reactions occurring with an epoxy ring preservation

Reactions without the epoxy ring occur with reagents that do not include an active hydrogen. Such reactions include those with phosphorus halides, sulfonic acid halides, carboxylic acid halides. Organophosphorus glycidol compounds are used as fire retardancy reactive agents to epoxy resins [3].



Scheme 2. Glycidol reactions occurring without an epoxy ring opening [3]

As a result of glycidol reactions with ketene or sodium acetate, glycidol acetate is created. Transesterification of glycidol with low-molecular carboxylic acid esters leads to the creation of suitable carboxylic acid glycidol esters. The addition of glycidol to cyanur chloride and acrylonitrile occurs easily, and the result is a triglycidol cyanurate and 3-glycidolpropionitrile. The first is used to give anti-crumpling properties to textiles, as well as for ageing on cellulose fibers. Furthermore, it is used as a component of varnish resins. The multifunctional tripolyglycidol cyanurate is obtained from polyglycidol and cyanur chloride. It is used in the production of lacquers, glues, elastomers and foaming agents. Moreover, these compounds are used for hardening gel photographic layers [3].

As a result of the alkyl vinyl ether's reaction with glycidol, a 2,3-epoxypropyl-(1-alkoxy)ethyl ether is created. Tetraglycidol silanes and alkyl-glycidol silanes are obtained as a result of the reaction of glycidol with tetraalkoxysilanes and alkylchlorosilanes. It is used in the production and modification of epoxy resins' properties, and as an adhesive agent in plastics strengthened with glass fiber [3]. In reactions with isocyanates, glycidol carbamates are created.

Glycidol in a surfactant synthesis

C10, C12, C14, C16, C18 maleic acid and fatty acid monoesters which undergo reactions with glycidol create a new group of non-ionic surfactants:

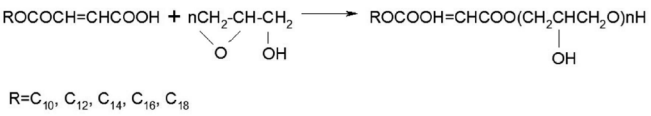


Fig. 4. The forming of nonionic surface active compounds with participation of glycidol [7]

The values of the critical micelle concentration, surface tensioning, foaming power, foam stability index and cloud point make it possible to deem them as very good washing agents and detergents [7].

Also ionic surfactants have been obtained from glycidol. Anionic compounds with a formula of $RO(CH_2CHOHCH_2O)_nCOCH{=}CHCOOM$ (n=1-100, M-H, NH_4^+, alkaline metal,

mono-, di- or triethanolamine cations, R-alkyl or alkylaryl) were obtained as a result of the glycidol's activity on alcohols or alkylphenols and the esterification of the half-finished product with a maleic anhydrate. The created monoesters were neutralized by a hydroxide of the alkaline metal, ammonium hydroxide or ethanolamine hydroxide [8]. This group may also include alkaline salts of alkylglycerol ethers, obtained from the reaction of glycidol with C_{10}-C_{20} alcohols. Products of oxyalkylining with glycidol were then sulfated with chlorosulfonic acid or a dioxane-SO_3 complex, and were neutralized.

Thermally susceptible reactive glycidol polyethers

A classic polymerization of glycidol leads to obtaining highly branched polymers, characterized by a difficult to control structure. Polymers of this type have a small usable significance, thus interest in them is small [9]. Mastering linear syntheses of glycidol polymers with a retention of the hydroxyl group, especially of

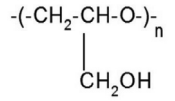


Fig. 5. Linear polymers of glycidol with retaining of hydroxyl group [9]

a "live" synthesis structure, has directed attention to the possibility of their use as emulsifiers and reactive polyethers susceptible to environmental stimuli. Because of this, an important value is that of the bottom critical temperature of glycidol polymers' and copolymers' solubility in water, whose criterion is the cloud point. Its value depends on the polymer's molar mass, its concentration in water, the number of hy-

drophilic and hydrophobic groups, the way of their binding, as well as other factors.

The condition which decides about the potential use of thermally susceptive polymers is the exact regulation of the temperature of the phase transition that accompanies their reaction to external stimuli. This temperature depends on the way of binding and fraction of hydrophilic and hydrophobic fragments in the polymer.

In order to acquire knowledge about these dependencies, linear copolymers (random (I) and block (II)), branched copolymers (comb-shaped (III) and hyperbranched/dendrimeric (IV)), as well as network copolymers (V) have been synthesized [9].

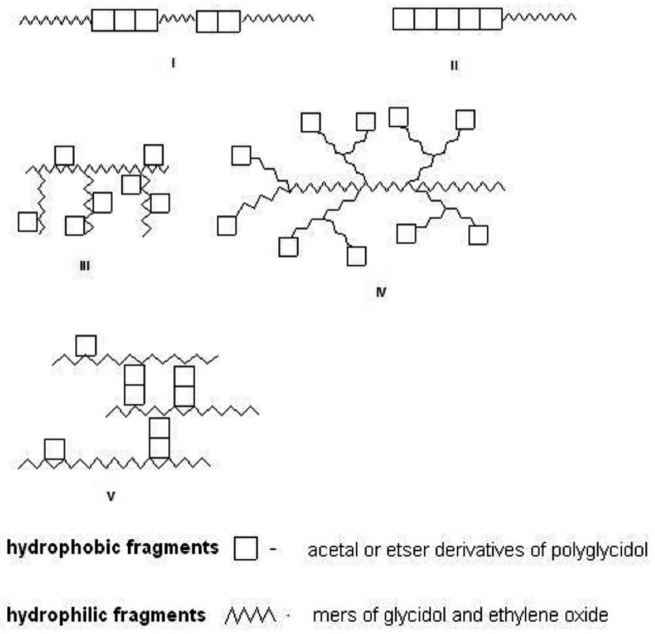


Fig. 6. Linear copolymers (disorganized (I) and block (II)), branched (comb (III) and hiperbranched/dendrometric (IV)) and cross-linking (V) [9]

The synthesis of glycidol's linear polymers requires the blocking of its hydroxyl groups. The blocking group has to be durable in conditions of anionic polymerization and easy to remove after polymerization, without damaging the main polymer chain.

An easy method of protecting hydroxyl groups is to carry out the reactions in acetal. In the case of glycidol and ethyl-vinyl ether, the course of the reaction is as follows:

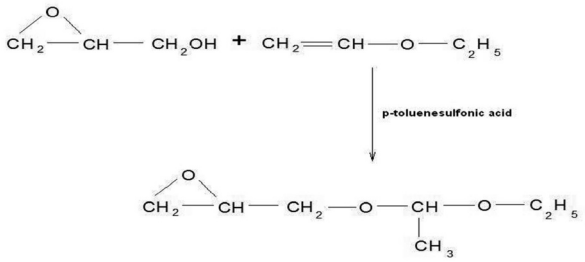


Fig. 7. Formation of 2,3-epoxypropyl-(1-ethoxy)ethyl ether (acetal of glycidol) [9]

The obtained 2,3-epoxypropyl-(1-etoxy)ethyl ether (glycidol acetal) is durable in conditions of an anionic polymerization, while acetal groups may be split off by means of hydrolysis in an acidic environment:

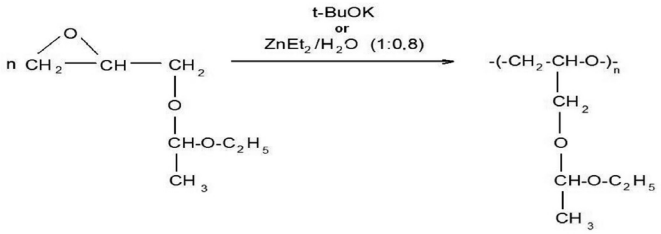


Fig. 8. Anionic polymerization of 2,3-epoxypropyl-(1-ethoxy)ethyl ether [9]

A glycidol homopolymer with a comb-shaped structure has been obtained via a "grafting" method by using a partly ionized polyglycidol to initiate the glycidol acetal polymerization. After removing the protective acetal groups, polyglycidol-*graft*-polyglycidol is obtained.

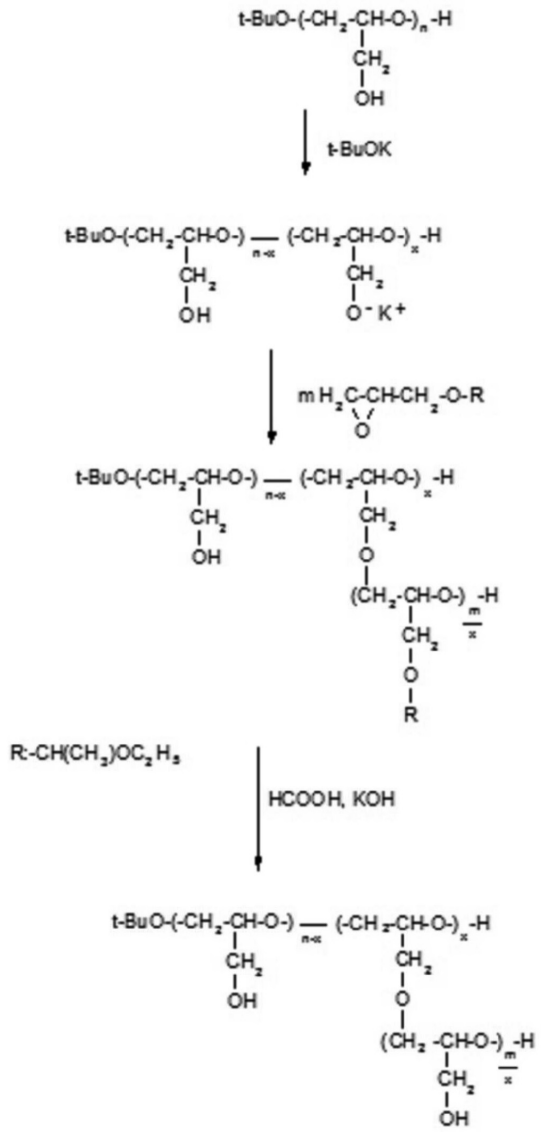


Fig. 9. Synthesis of glycodol homopolymer with comb structure [9]

The live character of the glycidol acetal's polymerization allows for simple syntheses of block copolymers containing glycidol. The polymerization of glycidol acetal was initiated with polyoxyethylene cesium glycolates and hydrophobic blocks of glycidol acetal polymers were added at the end of the chain [9, 10]:

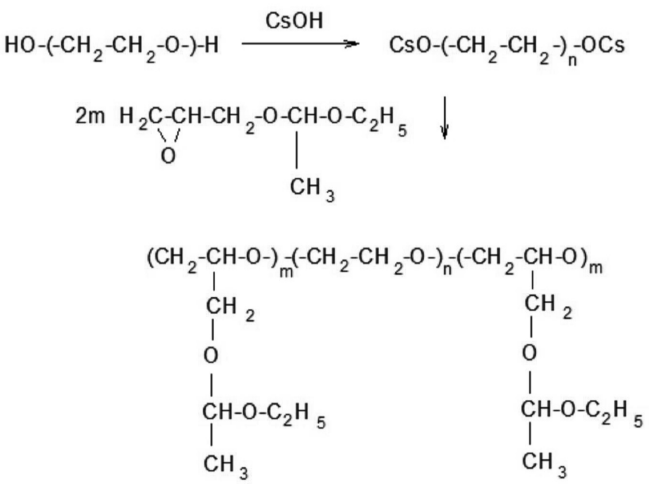


Fig. 10. Synthesis of copolymers with glycidol [9]

By changing the length of hydrophobic and hydrophilic blocks (ratio of the hydrophilic part to the hydrophobic one), copolymers were obtained which differ in the bottom critical solubility temperatures.

Glycidol in medicine

The use of a nonracemic glycidol in medicine may be arranged in four groups: cardiac agents, antibiotics, biochemical probes, pharmaceutics with different applications.

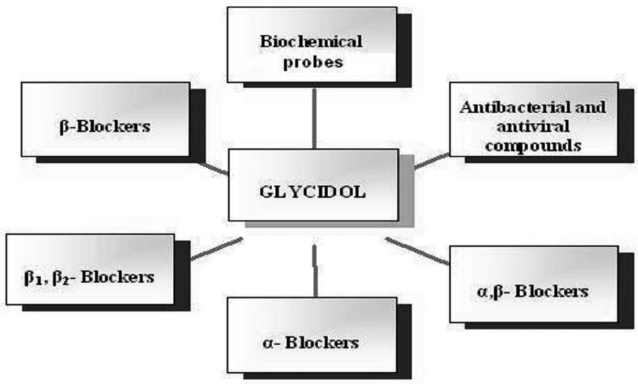
Glycidol is used in the production of cardiac drugs which lower high blood pressure, restore the heart rhythm (antiarrhythmitic) and improve the overall work of the heart muscle (Scheme 3). From the biochemical point of view, it is known that at least five mechanisms are related to this activity, out of which four depend on including the endocrine system. Such a system points to the existence of a link between the sympathetic nervous system and the involuntary muscles. Important neurotransmitters are adrenalin and noradrenalin, which are shown below [4].

Fig. 11. Chemical structures of adrenaline and noradrenaline [4]

Four receptors are known for these particles: α_1, α_2, β_1 and β_2. The α receptors are ascribed to noradrenalin in arteries and the heart muscle. They are responsible for maintaining a proper blood pressure. Especially α_1 receptors are postsynaptic and closely related to hypertension and arrhythmia. The α_2 receptor is characterized by a postsynaptic and presynaptic, non-specific effect, and its activity is generally linked to the side effects of drugs [4].

Blocking the β receptor system decreases the general activity of the nervous system. Compounds of a β-blocker character are used to increase the chances of survival after a heart attack. Two β receptors are known: β_1 and β_2. β compounds are especially important potential cardiac drugs. They influence the smooth muscles of the respiratory system [4].

A series of cardiac drugs derived from glycidol will be presented in the following order: β-blockers, β_1, β_2-selective blockers, α- or linked α/β-blockers and drugs with an adrenergic effect.



Scheme 3. The use of glycidol in obtaining cardiac drugs [4]

Generally, β-blockers are obtained from nonracemic glycidols [4]:

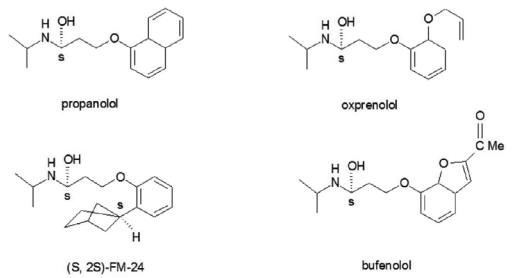


Fig. 12. Chemical structures of nonracemic glycidols [4]

By introducing amine to the nonracemic glycidol-aryl ether, the following compounds were obtained

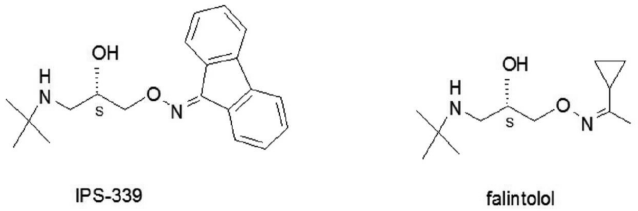


Fig. 13. Chemical structures of IPS-339 and falintolol [4]

Aryl ethers may be obtained through various ways, also with a microbiological oxidizing of allyl-glycidol ether and a nucleophilic substitution of glycidol.

Out of the two enantiomers of these compounds (fig. 13), with the exception of the last two, the enantiomer S whose structure is similar to the adrenaline structure, is the most active. Both enantiomers show the same activity [4].

β-blockers coming from nonracemic glycidols have the following structure (Fig.14)

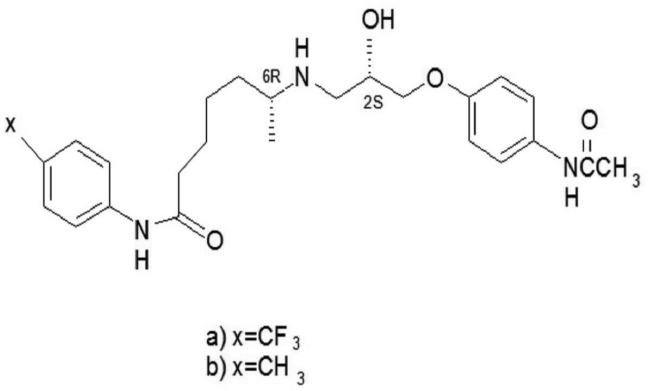


Fig. 14. Structure of ß-blockers coming from nonracemic glycidols [4]

The S enantiomer, whose structure is similar to the structure of adrenaline, has turned out to be more active. With relation to the two above compounds, the second stereocenter marked as 6 influences the effect of the drug marked as β_2 [4].

Some compounds obtained from nonracemic glycidol, which were then tested for α activity as potential hypertension and antiarrhythmic drugs, were presented below in Figures 15 and 16.

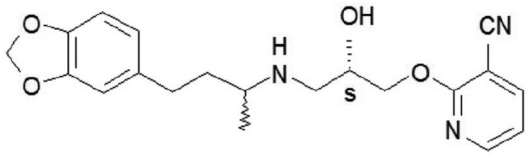


Fig. 15. Potential drug against hypertension and arrhythmia [4]

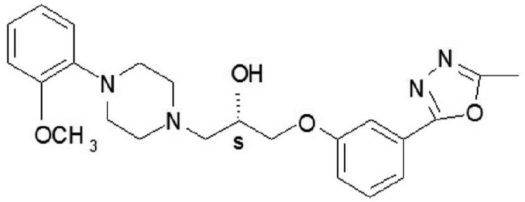
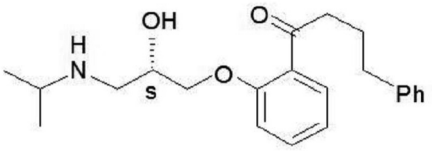


Fig. 16. Potential drug against hypertension and arrhythmia [4]

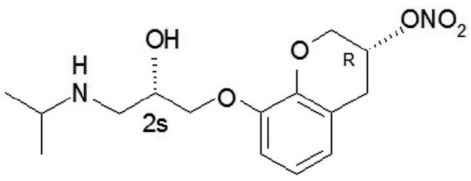
It is worth noting that the α-adrenenergic activity is fundamentally independent from the absolute configuration. Thanks to this, both these enantiomers have a similar effect with this receptor. A different activity of both enantiomers may have practical significance. In the case of the relation shown in Figure 17, the enantiomer R with a lower β activity is used in the production of hypertension-lowering drugs.



propafenone

Fig. 17. Chemical structure of propafenone molecule [4]

In the case of nipradilol:

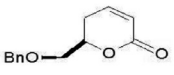


nipradilol

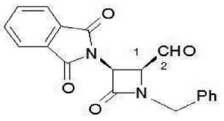
Fig. 18. Chemical structure of nipradilol molecule [4]

The second stereocenter has a decisive influence on the α activity and has a direct influence on the transport of K^+ ions. Two stereocenters act in each case, independently from each other. The stereocenter marked as R is important for the entire activity. The SR isomer is more active than RS [4].

From nonracemic glycidols it is possible to obtain antibacterial and potentially antiviral compounds, presented in Figures 19 and 20.

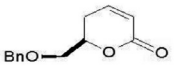


antibacterial antibiotic
Bn-benzyl

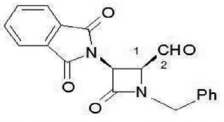


potential antibiotic half-product

Fig. 19. Chemical structures of compounds with potential antibacterial and antiviral effect [4]



antibacterial antibiotic
Bn-benzyl



potential antibiotic half-product

Fig. 20. Chemical structures of compounds with potential antibacterial and antiviral effect [4]

Many compounds coming from a nonracemic glycidol are not applied as drugs; they have been used as probes in order to explain the functioning mechanism of enzymatic systems. One example is the use of a nonracemic glycidol in the synthesis of glycerophosphocholines (PC).Glycerophosphocholines, obtained with the use of a nonracemic glycidol, have been presented in Table I [4].

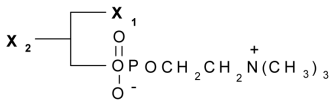
The compound presented in position 1 in Table I shows a high antineoplastic activity. Compounds marked as 3 and 4 are analogs of methyl ethers, containing an acetate group in position 2. They are known also as blood-forming agents. Positions 5-11 present a group of derivative glycerophosphocholines. Thioesters and thioethers in positions 12-14 have been used as substrates in obtaining A_1 and A_2 [4] phospholipase [4].

First of the amino acids (shown in Figure 21) has been used in studying β-lactam biosynthesis. The next two salts have been used in studies of a biosynthesis of aspartic acid and the metabolism of L-valine. The last of the amino acids was synthesized as a potential ligand in order to isolate the enzyme related to ethylene biosynthesis [4].

Glycidol-carbohydrate hybrids: a new family of DNA-alkylation factors

Table I

Glycerophosphocholines obtained with the use of a nonracemic glycidol [4]



	X_2	X_1
1.	CH_3O	$O(CH_2)_{15}CH_3$
2.	CH_3O	$O(CH_2)_{17}CH_3$
3.	CH_3CO (=O)	$O(CH_2)_{15}CH_3$
4.	CH_3CO (=O)	$O(CH_2)_{17}CH_3$
5.	CH_3CH_2CO (=O)	$O(CH_2)_{17}CH_3$
6.	$CH_3CH_2CH_2CO$ (=O)	$O(CH_2)_{17}CH_3$
7.	$CH_3(CH_2)_{14}CO$ (=O)	$O(CH_2)_{15}CH_3$
8.	$O(CH_2)_{17}CH_3$	CH_3CO (=O)
9.	$O(CH_2)_{15}CH_3$	$CH_3(CH_2)_{14}CO$ (=O)
10.	$O(CH_2)_9CH_3$	$CH_3(CH_2)_{16}CO$ (=O)
11.	$CH_3(CH_2)_{16}CO$ (=O)	$CH_3(CH_2)_{16}CO$ (=O)
12.	$CH_3(CH_2)_8CO$ (=O)	$CH_3(CH_2)_{11}S$
13.	$CH_3(CH_2)_8CO$ (=O)	$CH_3(CH_2)_8CS$ (=O)
14.	$CH_3(CH_2)_8CS$ (=O)	$CH_3(CH_2)_8CS$ (=O)

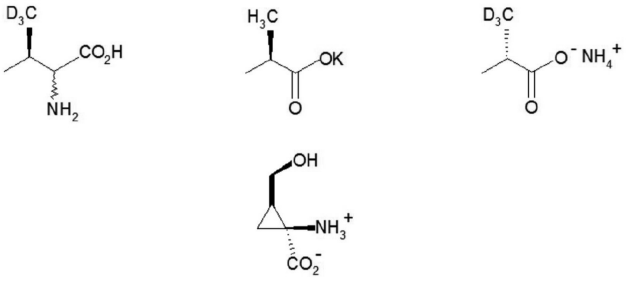


Fig. 21. Chemical structures of amino acids and salts [4]

Chiral glycidol was discovered in antineoplastic DNA-alkylation antibiotics, such as pluramycin, as well as in interactive carcinogenic DNA substances like B_1 aflatoxin oxide. These compounds belong to glycidol-carbohydrate hybrids which are an example of DNA-alkylation compounds composed only from an allyl and carbohydrate group. The structures of these compounds were presented in Figure 22.

Glycidol-carbohydrate hybrids 3 and 4 are epimers (a pair of diastereoisometric aldoses differing from each other only in the configuration around carbons C_1 and C_2) 5 and 6, while α-glycosides 3 and 5 are anomers (two isomers with a cyclic structure differing from each other in the location of the hydroxyl group at C_1 in aldoses or at C_2 in ketoses) of β-glycosides 4 and 6. It has been proved that these hybrids alkylate DNA selectively in place of N7 guanine and cause the fission of DNA [11].

The course of the glycidol-carbohydrate hybrids 3 and 4 has been presented in

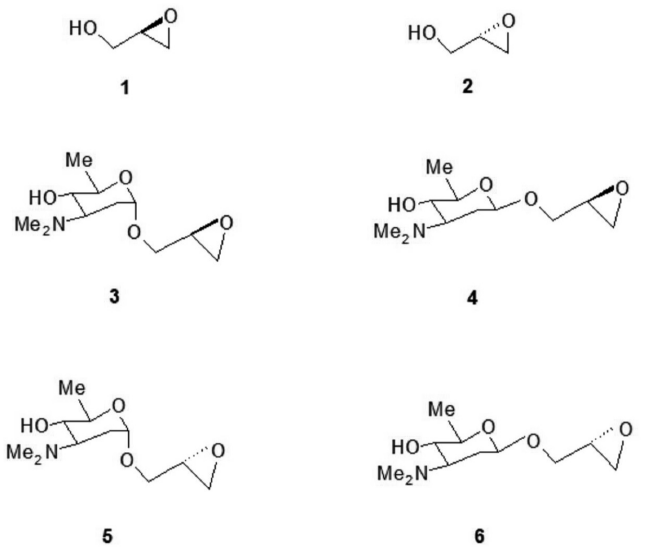
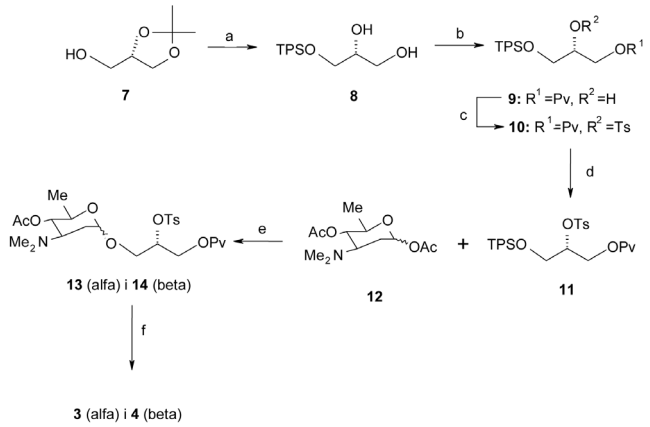


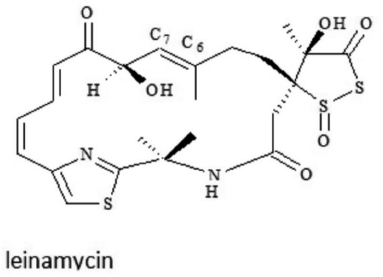
Fig. 22. Glycidol-carbohydrate hybrids – examples of compounds which can alkilate DNA [11]



Scheme 4. Synthesis of structures 3 and 4. Reagents and conditions: (b) PvCl (pivaline chloride), Et3N, CH2Cl2, 35°C, 7h, 96%; (c) TsCl (tosyl chloride), DMAP (4-dimethylaminopyridine), 60°C, 14h, 72%; (d) HCl, MeOH, 40°C, 12h, 72%; (e) TMSOTf (trifluoromethanesulfonic trimethylsilane), MS (methylsilane) 4A, CH2Cl2, 0°C, 0.5h, 84% (13/14=4/1); (f) MeONa, MeOH, 25°C, 5h, 52% for 3, 59% for 4; Pv- pivaline group, Ts- p-toluenosulfonyl [11]

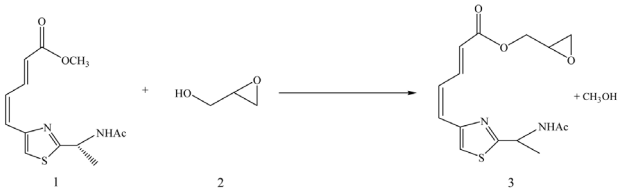
The initial compound in obtaining structures 3 and 4 is t-butyldiphenylsilyl (TPS), protected by compound no. 8. Hydroxyl groups in this compound have been selectively protected by a pivaline group (Pv), while secondary hydroxyl groups have been protected by p-toluenesulfonyl (Ts). These are paths 9 and 10 in the Scheme 4. After removing the TPS group in acidic conditions, the created alcohol underwent glycosidase with glycoside acetate 12 in the presence of TMSOTf which fulfills the role of an activator. This way, α- and β-glycosides 13 and 14 were obtained. The processing of isolated glycosides 13 and 14 with the use of MeONa causes the creation of epoxide with unprotected acetyl groups. The remaining hybrids 5 and 6 were obtained from the enantiomer 7 in a similar way [11].

Another compound that, besides the capability to alkylate DNA, also shows an antineoplastic activity, is *leinamycin* (Polish name) [12]:



Rys. 23. Chemical structure of *leinamycin* molecule [12]

This compound is bound by a double DNA spiral. This is interesting because *leinamycin* does not have functional groups to bind with DNA. A hypothesis has been put forward that the acyclic form of 5-(4-thiazolyl)-penta-2,4-dienone (3), situated on the left side of the *leinamycin* particle, is responsible for creating bonds with DNA. In order to confirm this hypothesis, a glycidol particle (2) has been introduced to diene (1) through an esterification substitution of the methyl group with a glycidol ligand (fig. 5) [12].

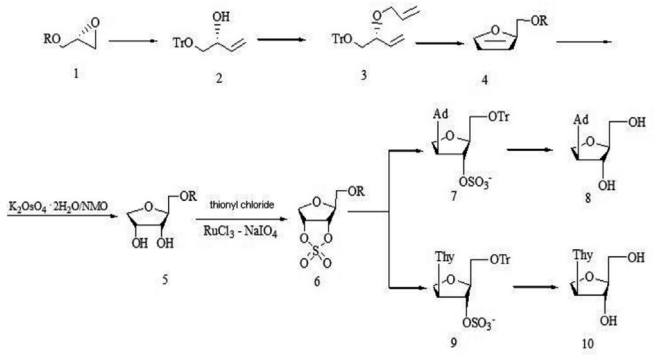


Scheme 5. Obtaining 5-(4-thiazolyl)-penta-2,4-dienone through an esterification substitution of the methyl group with a glycidol ligand [12]

Glycidol in the synthesis of antiviral drugs

One of the most important applications of glycidol is the synthesis of antiviral drugs. This group encompasses compounds active in the battle against HIV. Most often, they have been obtained from live organisms which limited access and had an influence on the high price.

(S)-Glycidol is **a substrate in the synthesis of L-isonucleosides** which belong to a group of biologically active compounds combining a selective activity against HIV and HSV with a high stability during acidic and enzymatic degradation. The scheme of obtaining L-isonucleosides has been presented in Scheme 6.



Scheme 6. Obtaining L-isonucleosides from (S)-glycidol [13]

The synthesis of L-isonucleosides occurs in two stages. In the first stage, the dihydrofuran particle is created (4). In the second stage, L-nucleosides are created (8,10). The creation of dihydrofuran will be initiated by a reaction whose aim is to protect the hydroxyl group in the (S)-glycidol particle (1) from further reactions. As a result of a further reaction with sulfur ylide (CH_2=SMe_2), the breaking of the epoxy ring occurs, together with the creation of butenodiol (2). The alkylation reaction with allyl bromide in a base environment produces the compound (3) which, after undergoing a reaction with

$RuCl_2(CHC_6H_5)$-$[P(C_6H_{11})_3]_2$, creates an intermediary product – a derivative of dihydrofuran with an efficiency of 78%.

The obtaining of L-isonucleosides (8,10) occurs through a series of further reactions. The first one is the reduction of dihydrofuran with a potassium salt hydrate of osmic acid (IV) in N-methylmorphine oxide ($K_2OsO_4 \cdot 2H_2O$/NMO), the result of which is 1,4-anhydroalditol (5) in the cis:trans proportion 1:4. A further reaction of the compound 5 with thionyl chloride, and oxidization with the use of $RuCl_3$-$NaIO_4$, leads to obtaining the compound 6. Reactions with adenine (Ad – purine base) and thymine (Thy – pyrimidine base) allow for obtaining compounds 7 and 9. The last stage is an acidic hydrolysis, the result of which is the creation of L-isonucleuosides (8, 10) [13].

Conclusions

The numerous applications of glycidol presented in the article prove how important working out a modern "ecological" technology of producing this compound is.

All the more so because in Poland this compound is not produced at all. One such method may be the epoxidation of allyl alcohol on zeolite titanium-silicalite catalysts, with the use of an "ecological oxidizer"-hydrogen oxide.

English translation by the author

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