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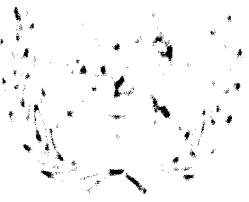
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STUDY OF THE PROBLEMS OF RURAL DEVELOPMENT

A. L. Saito  
Secretary, Regional Institute for  
Agricultural Research

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PAPER

## SUMMARY

### "NEW METHODS FOR THE PRODUCTION OF DIPHENYLPROPANE, DIVINYLBENZENE, GLYCIDOL AND SYNTHETIC GLYCERINE"<sup>1/</sup>

I. G. Pengailo  
USSR

#### Production of glycerine

A two-stage method for the production of synthetic glycerine from propylene oxide has been elaborated. First stage of the method: isomerization of propylene oxide to obtain allyl alcohol. The yield of allyl alcohol on the converted propylene oxide amounts to 89%.

Propylene oxide is isomerized to allyl alcohol over a lithium phosphate catalyst containing other ingredients. This catalyst provides for a conversion degree of propylene oxide up to 90%, the selectivity being in the range of 87 - 92%.

The catalyst capacity amounts to 700 - 800 g/l per hour.

Second stage of the method: synthesis of glycerine from allyl alcohol and hydrogen peroxide. As a result of the interaction of allyl alcohol with hydrogen peroxide in

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The amount of catalyst used at first attempt is determined which is enough to reduce the yield of product to 50%.

The yield of the product is then increased to 100% by the addition of more catalyst.

### Production of vinyl benzene

#### (1) Catalyst

This invention relates to a method of producing living benzene isomers and their blend, consisting of the following steps:

The following catalyst of the following kind is used:

1. Polyacrylate polymerized from diene, divinylbenzene;
2. Reductant, e.g., 10 mole % zinc + 10 mole % triethylbenzyl borane;
3. Polymerization agent, e.g., 10 mole % molybdate + 10 mole % divinylbenzene.

The invention is aimed at the production of high purity end products.

The cost of the process can be reduced over readily available commercial catalysts yielding similar amounts of material for the synthesis of divinylbenzene and similar isomers.

The apparatus used can be reduced in size of the catalyst-benzene reaction.

Due to the reduction of the raw material and energy consumption by 50% and 30% respectively and as a result of the decrease in capital investments and the cost of production per ton of divinylbenzene obtained by this method is 50% lower compared with 100% for divinylbenzene obtained by the acrylate polymerization.

### Production of high purity divinylbenzene

It is known that

it is difficult to obtain divinylbenzene from diacetylbenzene through trimerization of the latter with a primary or a secondary alcohol in the presence of a catalyst possessing both hydrogenating and dehydrogenating properties.

This invention concerns obtaining high-purity divinylbenzene.

In contrast to the previous methods, this one is based on the application of a new catalyst admitting both hydrogenating and dehydrogenating properties.

This method of making paper is a simple one, and it is not difficult to understand. In addition to the pulp, the paper-making process requires a certain amount of water, which is used to dilute the pulp and to help to separate the fibers. The water is heated to a temperature of about 100 degrees Celsius, and the pulp is added to the heated water. The mixture is then stirred until the fibers are completely separated.

### Production of Pulp and Paper

The production of pulp and paper involves several steps. First, the raw material, which is usually wood or other plant materials, is cut into small pieces. These pieces are then washed and cleaned to remove any dirt or debris. Next, the cleaned material is processed to remove any remaining bark or roots.

After the raw material has been prepared, it is pulped. This process involves breaking down the plant material into individual fibers. The pulp is then washed again to remove any remaining debris. Finally, the pulp is dried and formed into paper.

The production of pulp and paper is a complex process that requires a great deal of time and effort. The first step, which is the cutting and cleaning of the raw material, can take several hours. The second step, which is the pulping process, can also take several hours. The third step, which is the drying and forming of the paper, can take several more hours.

The cost of producing pulp and paper is relatively high. The cost of raw materials, such as wood, is the largest expense. The cost of labor and equipment is also significant. The cost of energy, such as electricity, is another factor that contributes to the high cost of production. The cost of producing pulp and paper is approximately \$100 per ton.

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the first time in history, the world's population has reached the point where there is no longer enough land available to support it. The result is that we must now turn to the sea for our food supply. This is a difficult task, but one that must be done if we are to survive as a species. We must learn to live in harmony with the natural world, and to respect the delicate balance that exists between all living things. Only then will we be able to ensure a sustainable future for ourselves and for generations to come.

The following are some of the more common catalysts of formic, oxalic and sulfuric acids, and the following list of acids, oil-soluble chemicals which can be used as diluents, and the polymerization of oil-based sulfonated amide resins can be conducted in the presence of such acids as sulfuric, citric, acetic, or phosphoric acid, and powdered aluminum, magnesium, calcium carbonate, and powdered aluminum. The same type of acid and styrene can be applied as synthetic tannins.



the catalyst is decomposed. It is also affected by various additives such as  
aliquat 336, triethylbenzyl ammonium bromide, tri-n-octylamine, bis-  
(tri-n-octylammonium) bromide, and tri-n-octylphosphine oxide. The  
additives are added to the polymerization mixture at the time of alkylation  
and are present during the polymerization reaction. The additives are  
present in amounts ranging from about 0.01% to about 10% based on the  
amount of alkylating agent.

the first time, the author has been able to construct equipment which can be used to measure the amount of chlorine available in the water. The results in Figure 1 show that the chlorine available in the water is abundant.

the first time, the author has been able to find a single example of a species which has been described as having a single dorsal fin, and which has been placed in the genus *Monopterus*. The author has examined the type specimen of *M. loricatus*, and has found that it has a single dorsal fin, and that the dorsal and anal fins are confluent. The author has also examined the type specimen of *M. albus*, and has found that it has a single dorsal fin, and that the dorsal and anal fins are confluent. The author has also examined the type specimen of *M. loricatus*, and has found that it has a single dorsal fin, and that the dorsal and anal fins are confluent. The author has also examined the type specimen of *M. albus*, and has found that it has a single dorsal fin, and that the dorsal and anal fins are confluent.

19. *Leucosia* *leucostoma* (Fabricius) *Leucosia* *leucostoma* (Fabricius) *Leucosia* *leucostoma* (Fabricius)

the absorption of light by the polymer is very substantial in the case of the aromatic polyesters. The absorption coefficient at 260 m $\mu$  is 10000 l.mole/cm<sup>2</sup> for the polyesters of phthalic anhydride and 10000 l.mole/cm<sup>2</sup> for the polyesters of terephthalic acid. The absorption coefficient at 260 m $\mu$  for the polyesters of phthalic anhydride is 1000 l.mole/cm<sup>2</sup> and for the polyesters of terephthalic acid 1000 l.mole/cm<sup>2</sup>. The absorption coefficient at 260 m $\mu$  for the polyesters of phthalic anhydride is 1000 l.mole/cm<sup>2</sup> and for the polyesters of terephthalic acid 1000 l.mole/cm<sup>2</sup>.

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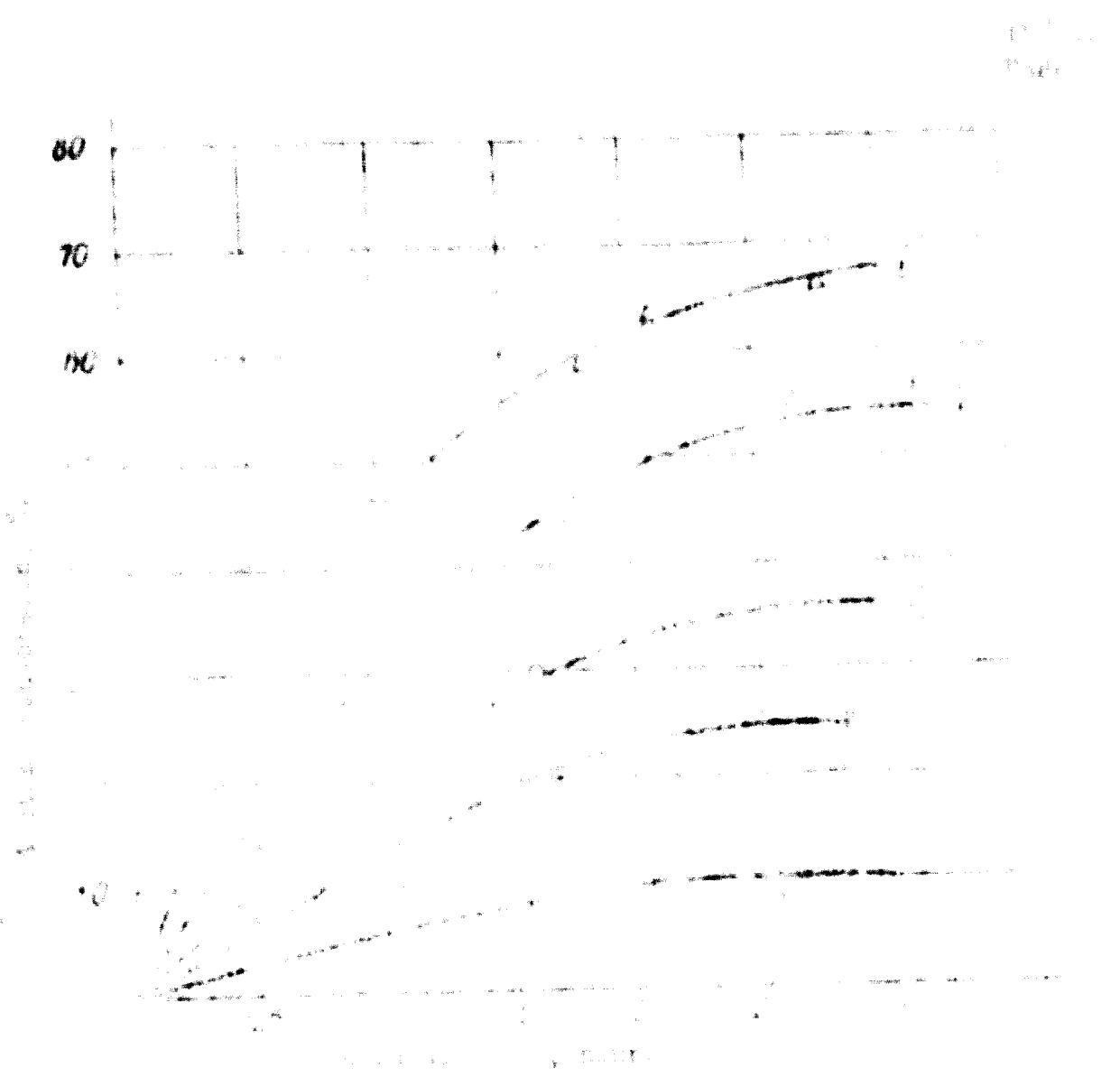


Fig. 1. Temperature vs. time for various initial temperatures and times.

time at which the temperature reaches 100°.

I.  $t = 0$ ,  $T_0 = 60^\circ\text{C}$

II.  $t = 0$ ,  $T_0 = 65^\circ\text{C}$

III.  $t = 0$ ,  $T_0 = 70^\circ\text{C}$

IV.  $t = 0$ ,  $T_0 = 75^\circ\text{C}$

V.  $t = 0$ ,  $T_0 = 80^\circ\text{C}$

VI.  $t = 0$ ,  $T_0 = 85^\circ\text{C}$

VII.  $t = 0$ ,  $T_0 = 90^\circ\text{C}$

VIII.  $t = 0$ ,  $T_0 = 95^\circ\text{C}$

IX.  $t = 0$ ,  $T_0 = 100^\circ\text{C}$

X.  $t = 0$ ,  $T_0 = 100^\circ\text{C}$

Fig. 2. Polymerization

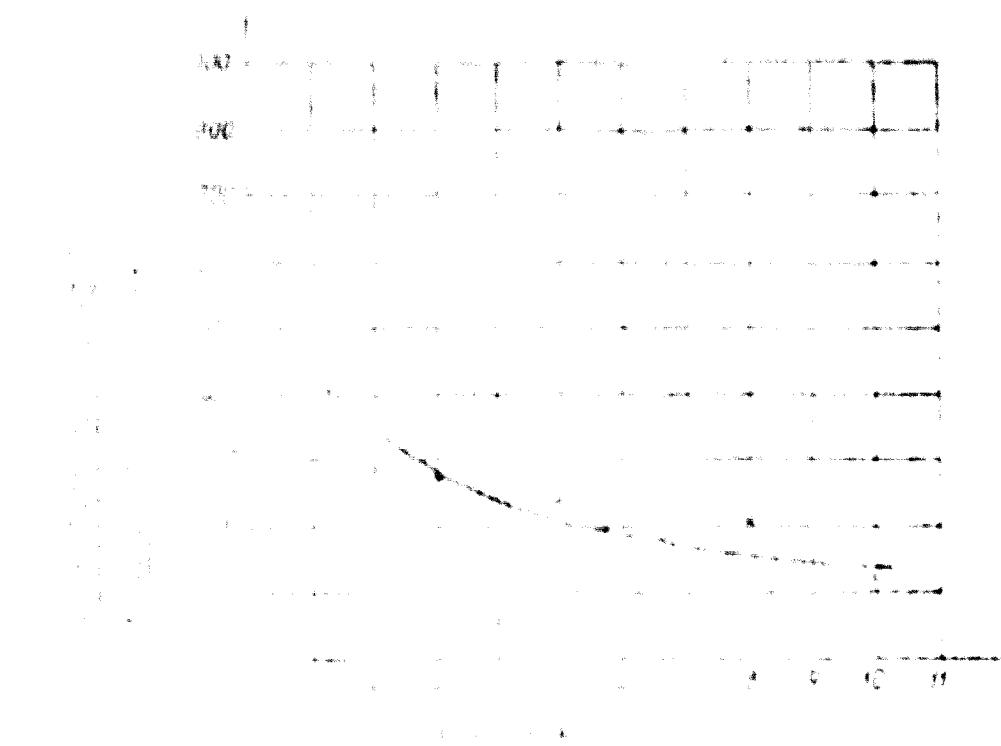


Fig. 2. Infrared spectrum of polymer with molar ratio of styrene : acrylonitrile = 1 : 1, temperature of the reaction = 75 °C,

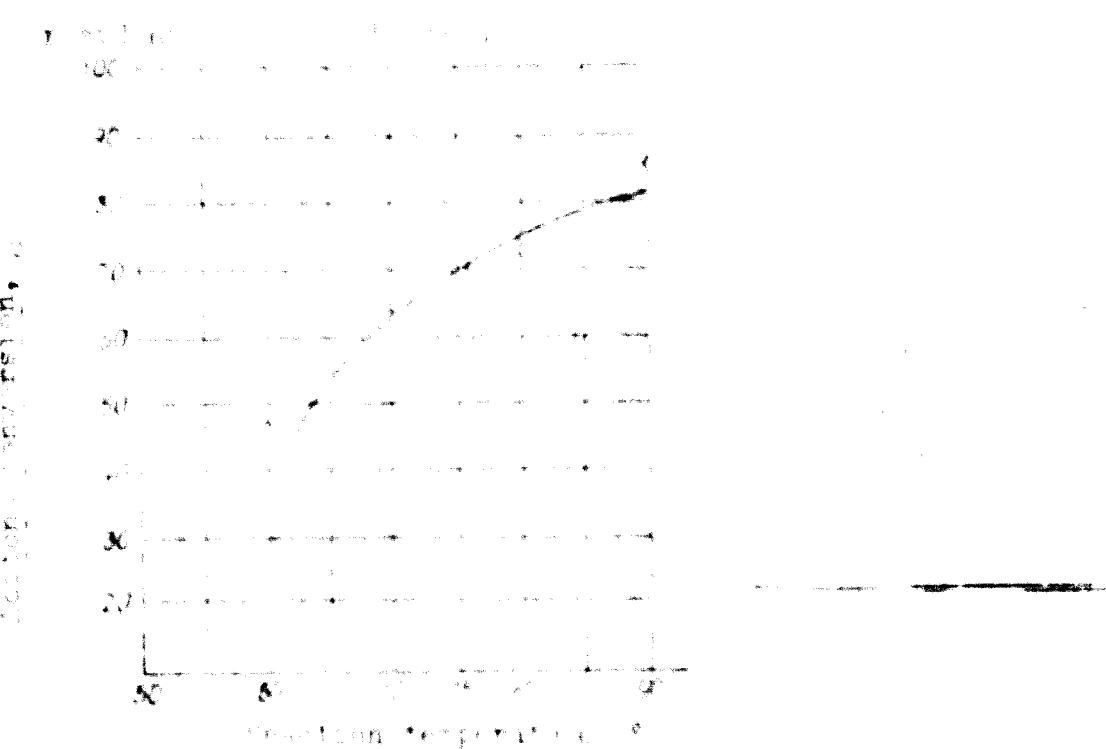


Fig. 3. Infrared spectrum of polymer with acetone  
concentration of 10% (v/v), molar ratio = 1, residence time = 1 hour.

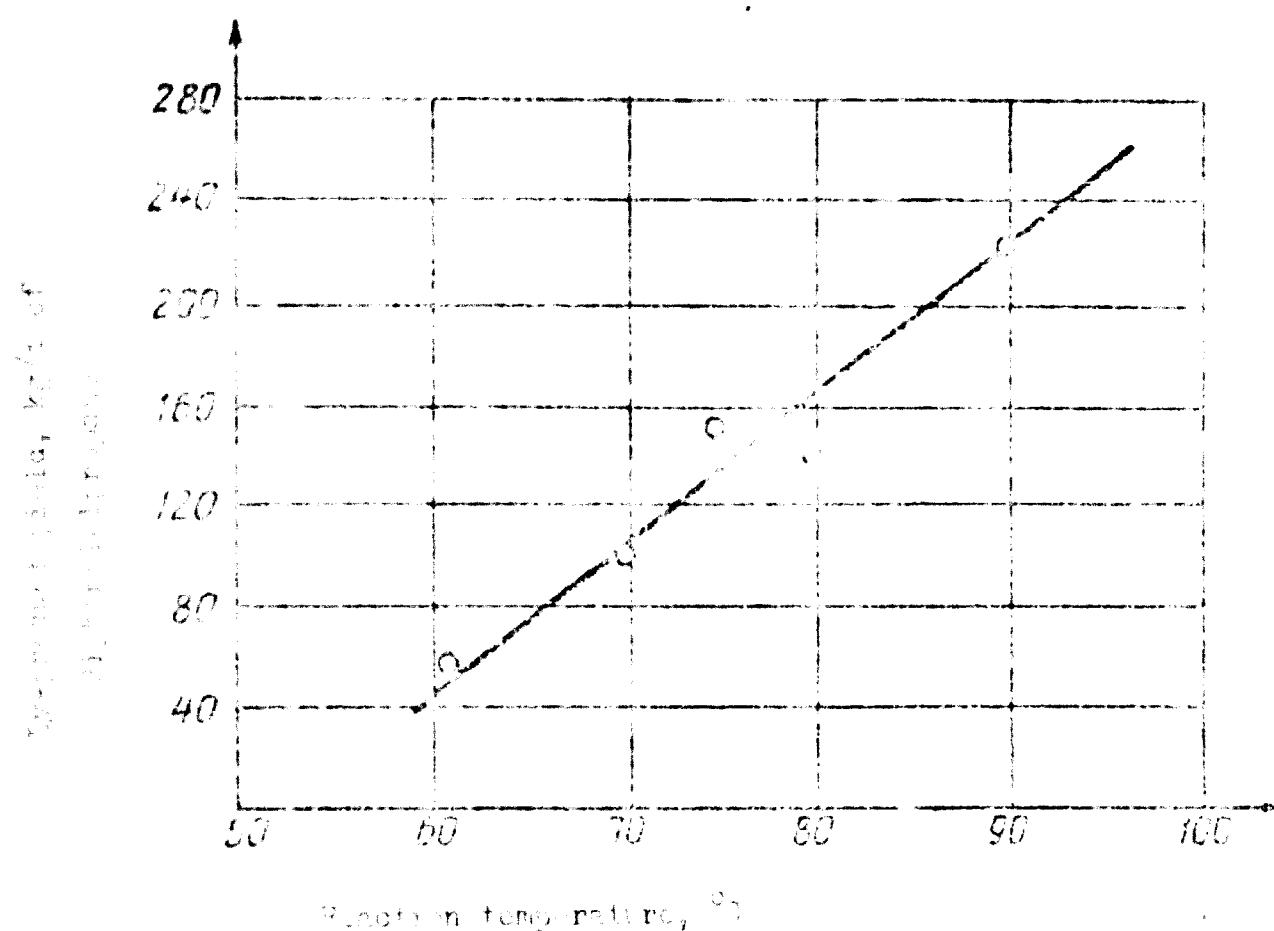


Fig. 4. Variation reaction temperature with by-product formation (molar ratio = 10 : 1, residence time = 2 hours).

Table 1: Productivity of the catalyst with additives.

Additive	Phenol to acetone molal ratio	Residence time, hours	Catalyst productivity, g/1.h.
N-2	5:1	0.48	455
	5:1	0.9	293
	5:1	1.8	170
	10:1	0.95	185
N-1	5:1	0.48	309
	5:1	0.95	194
	5:1	1.9	112

The yield of by-products may decrease greatly due to introduction of additives. For instance, the introduction of additive N-2 (phenol to acetone ratio in feed is 5:1) decreases the yield of by-products from 210 kg/t to 143 kg/t and to 113 kg/t of diphenylolpropane at F = 10:1. The diphenylolpropane produced in this case has high quality and properties as follows:

colour	- white
melting point	- 156.5 - 157 °C
content of volatile components	- not more 0.5%
optical density of alcohol solution measured with photocolorimeter -II-57	- 0.05
content of phenol	- not more 0.05%.

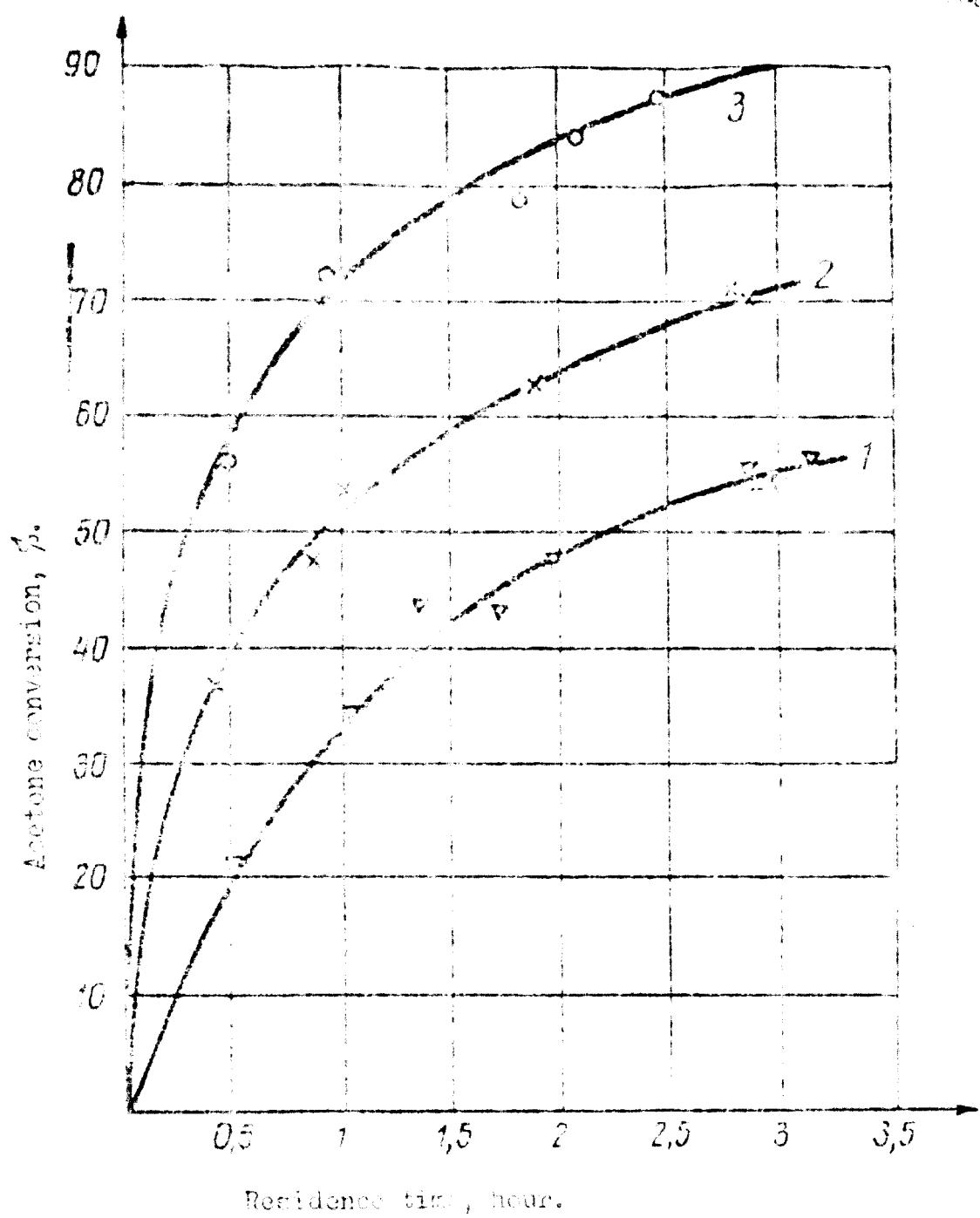


Fig. 5. Variation of acetone conversion with residence time  
at molal ratio of phenol to acetone in mixture of  
5 : 1,  $t = 75^\circ\text{C}$ .  
1. Without additive.  
2. With additive N 1.  
3. With additive N 2.

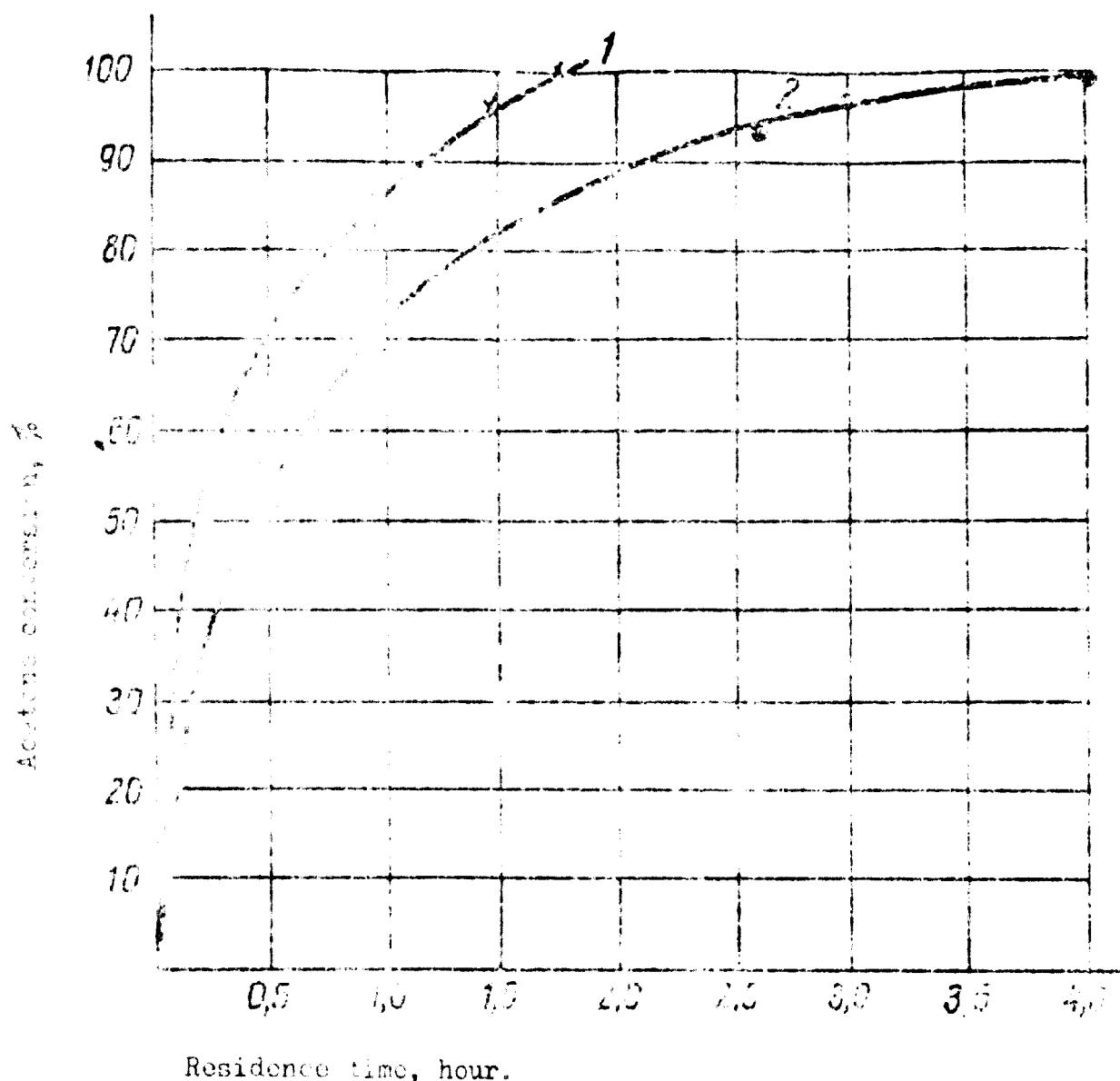


Fig. 6. Variation of acetone conversion with residence time for mixtures at molal ratios of 5 : 1 and 10 : 1 with additive N-2 at  $t = 75^{\circ}\text{C}$ .

1 - 10 : 1, 2 - 5 : 1.

Thus, the synthesis of diisobutylene over iron exchange resin with methanol gives the best yields of the hydrocarbons.

1. The use of a solid catalyst makes it possible to avoid the use of solvents, which are dangerous and difficult to handle electrically, and to reduce the cost of the plant.
2. Higher molecular weight hydrocarbons are received resulting in the removal of the separation equipment in the oldest type reactor.
3. The cost of labour is considerably reduced.
4. The quality of the product is improved.

According to our method diisobutylene can be synthesized over either exchange resin or the sulphuric acid catalyst and excess of phenol in comparison with its stoichiometric amount.

The 60-80% conversion rates of cyclohexane per pass and a catalyst regeneration time of 10 minutes.

The regeneration of the catalyst resin till regeneration requirement is not more than 100 hours.

The consumption rate of phenol and small because they can be reused. The catalyst regeneration is similar to that used. The sulphuric acid catalyst can be separated from impurities by crystallization or from solution. The flow diagram of the process is simple and it consists of this process will make a production of diisobutylene by continuous processing on a large scale through automation of the process as a whole.

At the moment, the construction cost and capital investments of the diisobutylene process will be much lower than in the case of production with the help of sulphuric acid and hydrogen chloride.

For instance, according to our method capital investments per unit are cut by 70% and construction cost per unit of labour hour in production with help of hydrogen chloride is 10%, so, in comparison with the sulphuric method, the savings obtained may be much more substantial.

## II. The production of meta- and para-divinylbenzenes of high quality.

Divinylbenzen is an important chemical used in organic synthesis.

Vinylbenzene can polymerize with other vinyl compounds to form comonomers of a different structure that can have wide applications.

Divinylbenzen is used mainly in the production of ion exchange resins. The structures of ion exchange resins can be synthesized as co-polymer of various monomers with divinylbenzen. These ion exchange resins are applied for complete demineralization and purification of water, for purifying oil, for removing metals from waste water, for deactivation of catalysts, as catalysts, in food and medical industries, etc. The production of such resins are applied on a commercial scale in the production of uranium from leach ores containing less than 0.5% of uranium.

The method of purification of antibiotics and other medicines with the help of ion exchange sieves is applied to a great extent.

The amount of vinylbenzene and in this case may be varied by variation of styrene to divinylbenzen ratio in the trifinal copolymer.

In the case of copolymer of styrene and divinylbenzen macro-porous ion exchangers, used for adsorption of organic compounds of higher molecular weight can be produced.

Divinylbenzen may be used as a main crosslinking agent in the preparation of various resins for special applications.

Resins produced by copolymerization of monomers with divinylbenzen are characterized by higher thermostability, lower solubility, good chemical stability and better insulating properties.

Thus, divinylbenzen may be used for improving the physical and chemical properties of plastics, rubber, man-made leather, organic diisocyanates, etc.

Divinylbenzen added at an amount of 0.5 parts by weight makes it easier to mold rubber in both calender and press because of decreased shrinkage of samples from 43 to 9%, swelling from 54% to 15% and also gives improved ageing resistance to the rubber.

Divinylbenzene is produced by a method of catalytic dehydrogenation of diethylbenzene in the presence of steam at a temperature of 600 - 650 °C which results in the formation of a mixture containing 15 - 25% of divinylbenzene and 75 - 85% of diethylbenzene, benzene, toluene, styrene, ethylvinylbenzene, etc. It is very difficult to separate pure divinylbenzene from this mixture because its components may form polymers readily. That is why, on a commercial scale, only a part of the divinylbenzene is rectified (up to 45 - 50%) in a complicated column.

American "Dow Chemical" and English "I.G." are producing 50 - 55% divinylbenzene maximum which contains, together with mixed divinylbenzene isomers, from 5 to 12 different compounds.

The method of catalytic dehydrogenation is characterized by substantial high consumption of starting diethylbenzene due to low selectivity of the process, lower content of divinylbenzene in mixture (15 - 25%) and troubles which occur in the course of divinylbenzene separation.

Investigations on creating an improved method of dehydrogenation of diethylbenzene to divinylbenzene have been carried out in various countries but, so far without any substantial results.

Divinylbenzene is produced in the course of a complicated reaction of catalytic dehydrogenation in two stages with the intermediate, ethylvinylbenzene, being formed while by-products are formed at each stage:

Diethylbenzene ----- ethylvinylbenzene ----- divinylbenzene.

Now it is not possible to separate divinylbenzene from by-products produced in the dehydrogenation of diethylbenzene by any economic method and this results in divinylbenzene of low quality and a high consumption of materials per ton of production.

For some years, Scientific Research Institute of Synthetic Alcohols and Organic Products has investigated a commercial method for the synthesis of divinylbenzene of high purity based on a new principle.

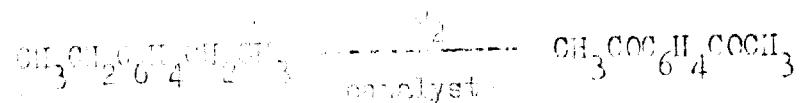
We are producing divinylbenzene by reactions with higher conversion and selectivity which permit either to avoid or to simplify the separation of compounds which polymerize and deteriorate readily.

This method provides for producing separated isomers of divinylbenzene of high purity.

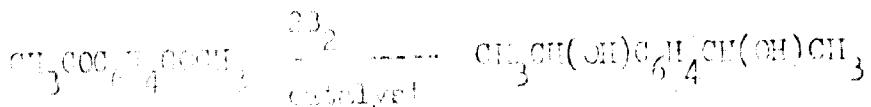
Diethylbenzene, formed together with ethylbenzene in the course of alkylation of benzene with ethylene in accordance with Friedel-Craft reaction in the presence of aluminum chloride as a catalyst is used in our process as a starting material. In this case, diethylbenzene is formed at an amount of 25 - 35% of ethylbenzene weight.

Divinylbenzene production according to our method may be described as follows:

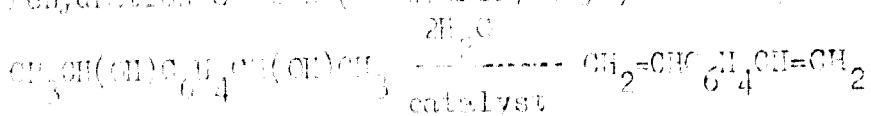
1. Oxidation of diethylbenzene to diacetylbenzene.



2. Reduction of diacetylbenzene to bis-( $\alpha$ -hydroxyethyl)-benzene



3. Dehydration of bis-( $\alpha$ -hydroxyethyl)-benzene to divinylbenzene.



Diethylbenzene is oxidized to diacetylbenzene in the liquid phase by oxygen or gas containing oxygen in the presence of salts of metals with transient valence (cobalt nitrate in the main). Diethylbenzene is oxidized to diacetylbenzene under mild conditions at a temperature 110-130 °C giving a high conversion of 70-75%.

We have investigated various methods of the reduction of diacetylbenzene to bis-( $\alpha$ -hydroxyethyl)-benzene such as Macrvein-Pondorf reaction in the presence of aluminum isopropylate catalyst and isopropyl alcohol donor, reduction with hydrogen over heterogeneous commercial catalyst (Barney nickel and nickel-chromium catalyst in suspended bed).

According to our method, diacetylbenzene is reduced with help of all said methods in such a selective way that compounds obtained after hydrogentation contain up to 91 - 99% of bis-( $\alpha$ -hydroxyethyl)-benzene and can be passed to next stage without additional purification and rectification.

We have investigated a method of hydrogenation of diacetylbenzene in liquid phase overnickel-chromium catalyst on fixed bed. However, this method gives a lower selectivity than in the case of hydrogenation of acetophenone, benzene and acetone, obtained in circulation. The content of diacetylbenzene in the product is found in circulation. The content of diacetylbenzene in the reaction mass obtained in the course of the hydrogenation of diacetylbenzene by a fixed bed is no more than 92 - 94%. The recovery of acetophenone + acetoxyacetophenone is necessary to obtain the reaction mass at a minimal pressure of 6.5 mm Hg.

Acetophenone + acetoxyacetophenone + divinylbenzene is dehydrated over magnesium sulphate which is due to very high selectivity of the reaction. Acetophenone + diacetophenone + the reaction mass product of the

Divinylbenzene can easily separated from impurities (of diacetophenone and 1,3-diphenylpropene) by simple distillation under vacuum.

Preparation of catalyst components for synthesis of concentrated divinylbenzene have been investigated and p-nitrophenol has been found to be best precursor. It is spent during thermal decomposition, living for 40 days at a temperature of 200°.

Method of gas chromatographic analysis which permit the determination of purity and composition of starting, intermediate and final products have been developed.

Samples of diacetophenone content have been tested successfully in the research investigation. Economic evaluation of various methods for the synthesis of divinylbenzene shows that according to our method the production cost and energy consumption are only half the projected values for the production of divinylbenzene by hydrocracking, and a quarter of the projected values in achieving commercial production of divinylbenzene.

Diethylbenzene, typical in the production of divinylbenzene is produced as a by-product in the course of alkylation of benzene with ethylene and consists of 60% of meta-isomer, 10% of para-isomer and 10% of ortho-isomer.

Relationship of ratio by ratio with the amount of theoretical plates as a given separation rate obtained in the course of computation has shown that rectification of starting diethylbenzene increases the production of

## EXPERIMENTAL

Commercially available benzene fraction rich in p-diethylbenzene only. This benzene was separated, distilled together with residual m-diethylbenzene, and the resulting mixture had increased boiling points of o- and p-diethylbenzene and decreased bp of m-diethylbenzene.

The benzene was separated from the p-isomer at the stage of distillation of the mixture. However, in this case diethylbenzene is separated from the mixture and different p-type ethylacetophenone is formed here. The benzene is obtained in the second stage ethylacetophenone is separated from the mixture.

Commercial benzene can be readily separated from mixed isomers by distillation. The fact is that p-diethylbenzene is a product of high volatility. The difference in boiling points of isomers is used here (bp of benzene =  $111^{\circ}\text{C}$ , m- $+32^{\circ}\text{C}$ , ortho-  $+39-40^{\circ}\text{C}$ ).

Commercial benzene is separated by the crystallization method and the benzene is purified from all of its impurities as ortho-phenylbenzene, m-phenylbenzene, and benzene.

Commercial benzene is separated by a combined method of distillation and crystallization. The benzene is dried and resublimation of the product is carried out in a vacuum.

The apparatus for the separation of diethylbenzene aimed at separation of m- and p-isomers. The separation of p-diethylbenzene is performed in a column consisting of two columns, the first one being for separation of m- and p-isomers. M-diethylbenzene is separated in the second column having the following parameters:

stationary phase	silica gel	187
bp of benzene	-	95
vacuum	-	200 mm Hg
boiling temperature	-	$124^{\circ}\text{C}$
boiling point of benzene	-	$165^{\circ}\text{C}$ .

2% pure m-diethylbenzene is obtained as a distillate and 26% m-diethylbenzene, 72% p-diethylbenzene and ortho-diethylbenzene - as a residue.

Part of diethylbenzene produced from pure 1,4-dioctylbenzene is oxidized first with sulfuric acid and then with dilute benzoyl peroxide, followed by subsequent reduction by the addition of the following reagents:

Aluminum chloride is added in excess. After hydrolysis of the mixed diethylbenzene and benzoyl peroxide, the resulting benzene is isolated by distillation. The remaining benzene is treated with concentrated sulfuric acid until no evolution of diethylbenzene is observed, upon heating.

The following products have been obtained with the following yields:

Precipitated 1,4-dioctylbenzene is obtained with a specific color which crystallizes at room temperature at 28-30°, refractive index  $n_{D}^{20}$  = 1.526. According to literature melting point is 31° and refractive index  $n_{D}^{20}$  = 1.526.

Melted 1,4-dioctylbenzene is a colorless liquid with specific color, refractive index  $n_{D}^{20}$  = 1.526. According to literature refractive index of m-divinylbenzene is  $n_{D}^{20}$  = 1.526.

A portion of the melt is solidified in a colorless figure with refractive index  $n_{D}^{20}$  = 1.526.

Thus, it may be concluded that the diethylbenzene produced in the method developed by the Research Institute for Synthetic Alkenes and Organic Chemicals is probably pure. The features are as follows:

1. Higher purity of final product which is about 95%.
2. Separation of catalyst from the mixed ones may be possible.
3. Better control of reaction and reduced consumption of starting diethylbenzene since from 1% to 1% is in the process of catalytic dehydrogenation of diethylbenzene to 2-t.

Propylene Oxide  
Report

Table 31

	theoretical yield	par- tial yield	ratio of partial to theoretical yield	commercial production
Commercial propylene oxide from propylene chloride and hydrogen chloride	1.720	4.76	10.0	
Commercial propylene oxide from propylene chloride and water	21.7	37.3	100.0	

III. The synthesis and properties of propylene oxide.

The synthesis of propylene oxide has been carried on very rapidly in 1938 and 1939. The first synthesis was obtained by heating allyl chloride with sodium hydroxide in the presence of potassium bromate.

Other methods of synthesis are available from propylene through acrolein and allyl alcohol.

It would be expected that formation of propylene oxide may have taken place during the synthesis of other organic compounds such as allyl phenyl ether, allyl phenyl sulfide, allyl phenyl hydrazine and cyclohexene oxide.

The following table is compiled from the literature for synthesis of propylene oxide from propylene chloride. The major portion of the material is probably from the chemical industry for the commercial synthesis of propylene oxide. The other large outlet is the production of allyl phenyl ether.

Propylene oxide is a allyl alcohol ester named in general as allyl oxide and is important to an increasing extent in the manufacture of plasticizers, rubberizers, fungicides, insecticides, adhesive membranes, glues, lubricating products, etc., for optical equipment, etc.

The following recommendations have been prepared for consideration by the  
magnetic tape committee. These recommendations are based on the information available  
in the present state of the art.

These recommendations are intended to provide a minimum standard for the use of magnetic  
tape media. They are not intended to be all-inclusive, but rather to provide a starting point for  
the development of a standard. The recommendations are as follows:

1. **Physical Characteristics:** The physical characteristics of the magnetic tape media shall be  
such that it can be handled and stored without damage. The tape shall be wound on a reel  
and shall be wound in such a manner as to prevent damage to the tape.
2. **Electrical Characteristics:** The electrical characteristics of the magnetic tape media shall be  
such that it can be read and written by standard magnetic tape drives. The tape shall be  
capable of being read and written at a rate of at least 1000 bits per second.
3. **Environmental Requirements:** The environmental requirements for the magnetic tape media  
shall be such that it can be used in a wide range of environments. The tape shall be  
capable of being used in temperatures ranging from -40°C to +80°C.

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such that it can be read and written by standard magnetic tape drives. The tape shall be  
capable of being read and written at a rate of at least 1000 bits per second.
3. **Environmental Requirements:** The environmental requirements for the magnetic tape media  
shall be such that it can be used in a wide range of environments. The tape shall be  
capable of being used in temperatures ranging from -40°C to +80°C.

The above recommendations are intended to provide a minimum standard for the use of magnetic  
tape media. They are not intended to be all-inclusive, but rather to provide a starting point for the development of a  
standard. The recommendations are as follows:

1. **Physical Characteristics:** The physical characteristics of the magnetic tape media shall be  
such that it can be handled and stored without damage. The tape shall be wound on a reel  
and shall be wound in such a manner as to prevent damage to the tape.
2. **Electrical Characteristics:** The electrical characteristics of the magnetic tape media shall be  
such that it can be read and written by standard magnetic tape drives. The tape shall be  
capable of being read and written at a rate of at least 1000 bits per second.
3. **Environmental Requirements:** The environmental requirements for the magnetic tape media  
shall be such that it can be used in a wide range of environments. The tape shall be  
capable of being used in temperatures ranging from -40°C to +80°C.

The second method is based on the application of complicated technology, the use of expensive catalysts and special materials used for the production of propylene oxide.

The third method is the so-called Synthetic Alcohols and Organic Acids method. This method consists of glycerine synthesis from propylene oxide. It has some advantages as compared with the first two methods: stabilized wastes and low

cost of production of propylene oxide.

In the case of the fourth method, the utilization of propylene the reaction of which with water should proceed in the presence of a catalyst. Our preliminary calculations show that it is a very promising one for the future.

### REFERENCES

Reference	Commercial production cost, \$
1	100
2	87
3	43

It is known that propylene oxide can be polymerized over such catalysts as aluminum chloride, boron trifluoride, etc. Among them only aluminum chloride gives a more selective way of isopropyl alcohol synthesis, but it can be deactivated

We have chosen a catalyst consisting of lithium phosphite combined with other components (up to 5%), which gave substantially increased service life and operating cycle for the catalyst.

This catalyst was tested for its service life on a laboratory and pilot scale and with the help of a copy of the reaction apparatus which we intended to apply in future for a commercial plant (tube of 3 m in height, catalyst volume = 720 ml).

In this case the products listed below were formed as by-products:

Propionic aldehyde	5.6 % by weight
Acetone	3.0 - " -
N-propanol	1.25 - " -
Isobutyl and non-identified compounds	1.15 - " -

Allyl alcohol of the following composition was formed:

Allyl alcohol	more than	93 % by weight
Propionic aldehyde		traces
Acetone		traces
N-propanol		1.5% by weight.

Consumption of raw materials per 1 t of 100% pure allyl alcohol:

1. propylene oxide ('00%) .....	1.14 t
2. catalyst .....	0.45 kg

#### Vestes.

100 kg of propionic aldehyde mixed with acetone are formed in the process (propionic aldehyde to acetone ratio is 2:1).

#### The production of glycidol and glycerine from allyl alcohol.

A method of the production of glycerine from allyl alcohol and hydrogen peroxide in the presence of tungstate catalysts including stages of separation and purification of the product, preparation and regeneration of the catalyst is investigated on a laboratory scale and then checked and tested on a pilot plant scale.

100% Acetone  
Tungsten

Initially in the presence of tungstic acid catalyst, 30% aqueous solution of allyl alcohol and hydrogen peroxide at a mole ratio of 1:1 and 60°C. was added to the reaction medium in the presence of tungstic acid 10 equivalents. After 1 hour the reaction medium

was allowed to stand until all unreacted allyl alcohol had reacted. The catalyst was removed from the reaction medium by a centrifuge and washed with acetone. The regeneration of the acid. Then the reaction mixture was separated and distilled. The yield of allyl glycerine was 60% and its composition with respect to the hydroperoxide was 10%. The yield and composition with respect to the hydrogen peroxide was 70%.

After the above results were obtained, allyl glycerine was tested on a pilot plant scale. The results obtained were in full analogy to those obtained in the laboratory design and for a continuous process.

After separation of the catalyst in the reactor, the catalyst is the viscosity of the reaction mixture is increased due to a solid residue which results in the formation of a thick slurry. The separation of the catalyst in the

reaction mixture is difficult. This is due to the fact that the catalyst leaves oxygen a new surface which is covered with a film of organic or other chemicals usually found in the reaction mixture.

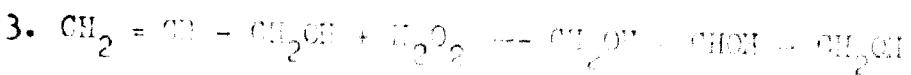
Catalyst can be easily separated from the reaction medium by a centrifuge and washed with acetone. After additional treatment. The application of the catalyst to the reaction mixture, the use of catalyst regeneration, the addition of a small amount of acetone, the addition of a sufficient amount of reagent and the addition of a small amount of water to the reaction mixture contaminated with organic material.

A method for the production of synthetic glycerine from allyl alcohol and hydrogen peroxide in the presence of our catalyst consists of stages as follows:

1. Reaction of allyl alcohol with hydrogen peroxide to form glycidol-glycerine mixture.
2. Recovery of unreacted allyl alcohol from the reaction medium and its purification.
3. Catalytic hydrolysis of the glycidol in the reaction media to obtain glycerine.
4. Concentration by evaporation of glycerine solution.

Allyl alcohol reacts with hydrogen peroxide in the presence of a heterogeneous catalyst. The epoxidation of allyl alcohol to glycidol proceeds, in general, under recommended conditions. Small amounts of glycerine (1 mole per 25 moles of glycidol) present in the reaction media are formed as a result of glycidol oxidation or a parallel reaction of hydroxylation of allyl alcohol to glycerine.

These reactions may be described by equations as follows:



In order to increase glycidol yield and avoid the side reactions of oxidation the reaction of epoxidation is preferably conducted under mild conditions such as a temperature below 20°C and at a selected reagent-to-reagent ratio.

Selectivity of the epoxidation increases as allyl alcohol to hydrogen peroxide molar ratio increases.

Glycidol is hydrated to glycerine directly in the reaction medium without separation.

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Glycidol reacts with water as follows:



Different catalysts will be used in the following for this reaction to determine which ones will produce at normal pressure and different temperatures the greatest of catalyst or without it.

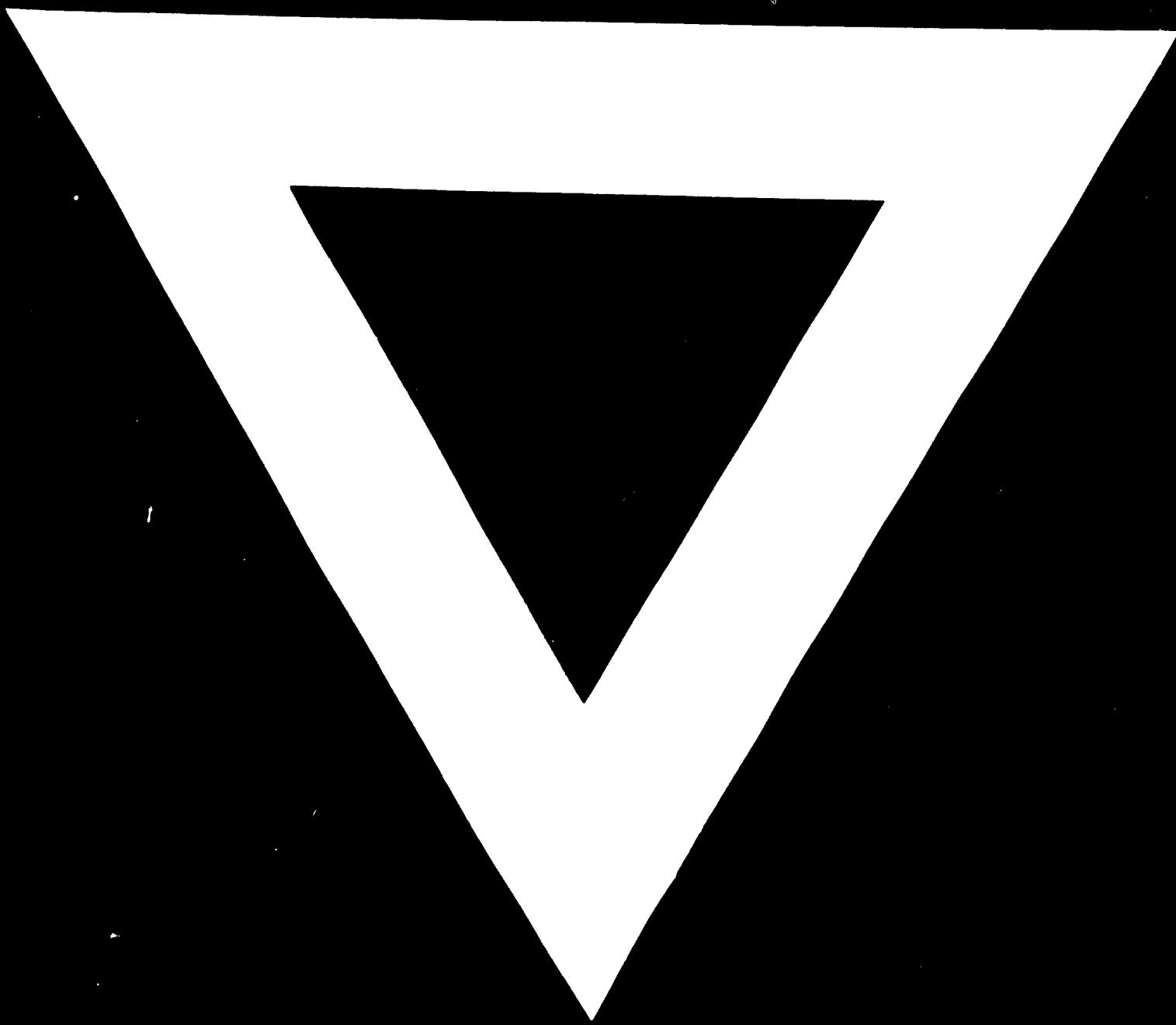
The following table gives the results of the ability to absorb 10% grade glycerine by various materials. The materials were purified the several methods in order

The optical properties of the polyesters prepared by this method are given in Table I.

Method of preparation of the distilled glycerine:

Consumption rate	unit	Consumption rate
kg	t	kg
0.77	t	0.45
2.0 - 1.0	kg	





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