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Obtaining the retainer for waterproofing road bitumens

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ABSTRACT: Introduction. Bitumen is a mixture of hydrocarbons and hetero-organic compounds. It is one of the most popular building materials today. Due to the growing consumption of bitumen for various purposes, the requirements for its quality characteristics are increasing, which prompts a comprehensive study of the physical and mechanical properties and methods of its modification. Currently, various additives are used, from inorganic materials to organic binders, including waste chemical, petrochemical and household industries. These additives create a nanodispersed structure inside the bitumen, which provides a change in the physical and mechanical properties in the required direction. **Methods and materials.** The work proposes a method for obtaining a fixer for waterproofing road bitumen based on nitrogen-containing organic compounds. The goal of research is to study the effect of the additive-derivative of triethylenediaminedicyan, which leads to the formation of a nanodispersed structure of bitumen of the "sol-gel" type, the quality indicators of which will meet the requirements of the new standard GOST 33133-2014 "Viscous road oil bitumen". The object of the study is the bitumen production unit of workshop No. 14 of Gazprom neftekhimSalavat LLC, designed to produce commercial bitumen: oil road grades CB 90/130 in accordance with GOST 22245-90, used in road, civil and industrial construction as a binder and waterproofing material. **Results and discussion.** In the course of the study, the nature of the interaction and the effect of the modifier on the properties of bitumen, which ensure the production of nanostructured bitumen of the "sol-gel" type, were revealed. As a result of the study, a comparative assessment of the effect of the fixer on the properties of waterproofing bitumen revealed a significant improvement in physical and mechanical properties in comparison with bitumen grade CB 90/130. **Conclusion.** The obtained compound based on triethylenediaminedicyancan can be used as a fixing additive to road bitumen.

KEYWORDS: bitumen, tar, asphaltenes, oils, dicyandiammonium compounds, nanodispersed systems, fixing additive, physicochemical properties.

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INTRODUCTION

The use of high quality petroleum bitumen ensures the durability of asphalt concrete pavements in conditions of heavy traffic. The quality of bitumen as oil dispersed systems is directly related to their structure and properties, which are determined by the quantitative ratio of oils, resins and asphaltenes [1–3]. These three main structural elements of bituminous binders interact with each other and form one or another kind of microheterogeneousnanodispersed structure [4–6].

The physical and mechanical properties of bitumen depend on the production technology and the nature of the feedstock. The raw material source for obtaining bitumen for various purposes is the residual products of oil refining. As practice shows, these residues are subjected to all kinds of modification methods in order to give them high quality characteristics. In connection with the increase in the pace of construction in our country, the consumption of bitumen continues to grow, so the production and improvement of their quality characteristics is relevant [7, 8].

Modification of bitumen contributes to the expansion of the temperature range of operability, increased heat and frost resistance, ensuring the reliability and durability of structures.

Obtaining high-melting bitumen by oxidizing raw materials does not allow achieving results in terms of quality that meet the requirements of GOST. Therefore, compounding is the most optimal method. To create high-melting compositions, it is necessary to select certain additives that act as astringents. Therefore, the first stage of the work was the study of the process of obtaining a binder additive [9–11].

METHODS AND MATERIALS

A method of obtaining a waterproofing composition by mixing bitumen, polymer additives and plasticizers, characterized in that the process takes place in mild conditions, the resulting bitumen is resistant at temperatures from minus 20 to plus 80°C. Known modifications with isoprene copolymers, oligomerizates. With the addition of binders, no more than 5–7% of 100% by weight of the waterproofing composition.

To increase adhesion, mastic is used, it additionally contains waste from the production of butadiene-styrene latex, with the following ratio of components,% wt.: bitumen 20–25%, asbestos 20–25%, waste from the production of butadiene-styrene latex 3–15%, sulfur sludge 2.0–5.5%, water – the rest. Various copolymers are used for modification that increase plasticity, reducing water absorption in the ratio of % wt: oil hydrocarbons 55–63%, resins 12–15%, asphaltenes 25–30%, and as an additive – atactic polypropylene or a concentrate of rubber isoprenes with bitumen in ratio 1:1 at the following ratio, % wt.: oxidized residue of atmospheric distillation of oil 85–95%, polymer additive 5–15%.

Bituminous-latex emulsion composition containing bitumen, an emulsifier based on sulfonal and synthetic fatty acids, which increases weather resistance, at the following ratio, wt%: bitumen 100%, vat residue of synthetic fatty acids 1–2%, emulsifier 2.8–5.1%, synthetic latex 65–70%, 45–48% solution of lignosulfonates 2–4%, hexamethyldisilazane 2–4%, water 55–65%.

In the work of obtaining a composition for waterproofing, the filler is polyethylene, a plasticizer, extracts (waste after selective purification of distillate oils, asbestos and polypropylene glycol. This composition is distinguished by high atmospheric resistance, the following ratio of components, wt%: polyethylene 1.0-2.2%, asbestos 17–30%, extract 3.0–5.5%, propylene glycol 0.2–1.5%, bitumen – the rest.

Bituminous composition for roofing and waterproofing materials, containing bitumen and a mineral filler, characterized in that in order to increase the biostability of the composition, as a filler it contains a cake of copper smelting production, with the following ratio of components, wt%: bitumen 75–90%, cake 10–25%.

It is known that styrene-butadiene rubbers are part of the bitumen as a binder. Their use makes it possible to improve the technical characteristics of the product, fillers for bitumen – asbestos, white spirit, rubber (styrene butadiene), oil (automobile) [12–14].

In order to increase the weather resistance, add to bitumen: polyethylene, oil (automobile), oligovinylethanolamine, talcomagnesite.

A method of obtaining waterproofing mastic. Mastic contains 39–99% wt oxidized bitumen and 1–8% oxidized polyethylene. To regulate the viscosity, up to 40% of a saturating substance is introduced into its composition – not oxidized bitumen. Mastics containing 71–89% of oxidized bitumen, 1–4% of polyethylene and 10–25% of a saturating substance have optimal properties; 50–60% of mineral chips are used as a filler. The mastic is prepared by adding oxidized bitumen to oxidized polyethylene. Polyethylene, molecular weight – 2000–6000, softening point 130–1500°C, Brookfield viscosity at 149°C 5000–30000 cP.

A waterproofing composition comprising bitumen, a thermoplastic copolymer of ethylene, propylene, ethylidene, characterized in that, in order to increase biostability, it contains a filler such as ash, asbestos, fiberglass, polymer fibers, soot of silicate or carbonate mineral powders, with the following composition ratio, wt%.: bitumen 20%, ternary copolymer and polyethylene 20%, filler 60% (polyethylene content is equal to 0.3% by weight of the ternary copolymer weight).

The method of obtaining a bitumen composition for priming steel pipes, which allows to increase the stability of the material, is used as a binder CPL (50:50). It is prepared by mixing at 200°C for 2 hours a mixture with the following content of components, wt%: bitumen 45%, a mixture of CPL 42%, 2-methyl-1-butene and 3-methyl-1-butene 42%, high density polyethylene 5%, isobutyl rubber 5%, epoxy 5%. Theflowcoatinghasahightearstrength.

Waterproofing material made of polymer bitumen, characterized in that the cover layer consists of a mixture of bitumen (penetration 70-420*0.1 mm, softening temperature 20–50°C, 10–30% homo- and copolymers based on atactic polypropylene, 5–20% isotactic polypropylene (melt index 20–40).The outer surface of the cover layer contains polypropylene crystals in the form of spherulites, which improve adhesion and prevent oil components from sweating out of bitumen.

Bitumen at a temperature of 160–200°C is mixed with polymers, the mixture is homogenized under high gravity, at 180–200°C it is cast in the form of a film and cooled at a rate of 120 K/min.

Waterproofing composition, characterized in that in order to increase the adhesion to concrete and metal, as well as to increase the temperature range, add to bitumen: polypropylene and plasticizers filtration waste. The sludge contains 30–50% dialkyl phthalates, 2–7% sodium salts of monoesters of phthalic acid, the rest is activated carbon. The proposed composition has a softening temperature by ring-and-ball method 92–100°C, heat resistance 85–95°C, adhesion to concrete and metal, respectively, 0.61–0.86 and 0.7–0.9 MPa, water absorption 0.8–0.83%.

Currently, many researchers make a great contribution to the selection of effective fillers, binders, etc., in order to impart crack resistance, strength, adhesion and other performance characteristics to bitumen in various climatic conditions [15]. When studying reagents that meet the requirements of GOST, we were guided by the operating conditions of the material and the design of the reaction unit, as well as climatic influences (Table 1).

These properties were the main criteria when choosing polymer additives for bitumen.

By the nature of the effect on bitumen, polyethylene and EPC belong to additives that change the structure of the product. Table 2 shows the composition of mastics based on polymer additives to bitumen.

Currently, there are GOSTs for bitumen, which are used as insulating bitumen. The characteristics of insulating bitumen are given in table 4 [16–17].

Synthesis of dicyandiammine is carried out in a reactor, which is a four-necked round-bottom flask equipped with a stirrer, thermometer, glycerin bath, separating funnel, reflux condenser, thermostat. 46 g of ethylenediamineare poured into the reactor, the temperature in the reactor is brought to 30°C and 0.23 g of the catalyst is tetraethylammoniumethoxide (alcohol solution). With stirring, 106.0 g of propenenitrile (acrylonitrile) is added dropwise to the reactor through a separating funnel and the mixture is stirred for two hours.

The reaction of the synthesis of triethylenediaminedicyan:

$$2CH_2 = CH{-}C \equiv N + H_2NCH_2{-}CH_2{-}NH_2 + \\ + [(C_2H_5)_4N]^+ \cdot O^-(C_2H_5) \rightarrow \\ N \equiv C{-}CH_2{-}CH_2{-}HN{-}CH_2{-}CH_2{-}NH{-} \\ {-}CH_2{-}CH_2{-}C \equiv N$$

Next, a solution of methanal (37% aqueous solution) was added to the reaction mixture, in a molar ratio to ethylenediamine equal to 1:2. At a temperature of 30°C, the mixture is stirred for two hours to obtain a compound of structure:

$$N \equiv C{-}CH_2{-}CH_2{-}N(CH_2OH){-}CH_2{-} \\ {-}CH_2{-}N(CH_2OH){-}CH_2{-}CH_2{-}C \equiv N.$$

Table 1

Basic technical requirements for waterproofing various structures

Materialproperties	Hydraulic-structures	Overground structures	Underground structures	Roofs
Waterabsorption, %wt.	1.0	3.0	1.5	5.0
Swelling,%vol.	0.5	1.0	0.8	1.5
Heat resistance, not less, °C	+40	+60	+40	+70
Brittlenesstemperature, °C	–15	–40	–5	–50
Tensile strength at break, N/m, MPa	1.0	0,8	0.5	1,5
Resistance against aggression: – Total acid, pH, less – Alkaline, g/l – Sulfate, mg/l	 5.0 80 2000	 4.0 100 5000	 4.0 150 2000	 6.5 50 1000
Decrease in elongation after 500 h, %	25	10	30	5
Minimumdurability, years	50	10	50	10

Table 2

Composition of mastics (polymer–bitumen), wt%

Component	Bitudien-3	Bitudien-L	Bitulen-90
Powderedpolyethylene	–	–	3
Polydiene	20	20	–
Petroleumbitumens for insulation of oil-and-gas pipelines: – BPI-IV – BPI-V	 – 80	 – 80	 97 –

Table 3
Main technical properties of bitumen-polymer mastics

Mastic	SPby ring-and-ball method, K, not less	Elongation, cm, notless	Penetration, 10^{-1} mm, not less	Maximum temperature of the transported product, K	Permissible ambient temperature when applying mastic, K
Bitudien 3	343	4	30	293	253–278
Bitudien-L	363	3	20	308	263–303
Bitulen-90	363	2	15	308	268–308

Table 4
Characteristics of insulating bitumen GOST 9812-61

Indicators	Insulating		
	BPI–IV	BPI–IV-3	BPI–V
1) Penetration$_{25,}$ 0.1 mm	25–40	30–40	not less 20
2) Elongation at 25°C, cm, notless	4	4	2
3) Softening point, °C, notless	75	65–75	90
4) Water resistance for 24 hours,% weight, no more	0.2	0.2	0.2
5) Content, %: а) water-soluble compounds, no more; б) asphaltogenic acids, not less; в) paraffin, no more; г) sulfur, nomore	 0.2 1.25 – –	 0.2 1.25 4 2	 0.2 1.25 – –

Ethanic acid is added dropwise to the resulting compound through a separating funnel (the molar ratio of ethanic acid to ethylenediamine is 2:1). Stirring is carried out at a temperature of 30÷40°C for at least three hours.

During the study, the following methods were used:

1. Compounding of additives is the mixing of different additives in different proportions in order to improve their effect on the properties of bitumen. Additives in different mass ratios were placed in a metal container, placed on a hotplate, a stirrer was installed and stirred for 15 minutes, then the stirrer was turned off, and a sample was taken to determine various properties. After that, the stirrer was turned on, and so every 15 minutes for two hours.

2. Compounding bitumen with additive

In a metal container, 100 g of bitumen was taken and, depending on the ratio, the additive, a stirrer was placed there and stirred for one hour, and a sample was taken for analysis, turning off the stirrer.

3. Softening temperature according to GOST 11506-73

The temperature at which the bitumen in the ring of given dimensions softens and, moves under the weight of the ball, touches the disk of the device. Pour bitumen with some excess into two rings, which are preheated. After cooling the rings with bitumen, smoothly cut off the excess with a heated knife flush with the edges of the rings. Install a thermometer in the intermediate hole. Bitumen research is carried out in a glycerin bath at a temperature of 33–35°C. After 10 minutes, the suspension is removed, a heated steel ball is placed with tweezers, and placed back into the bath, set so that the plane of the rings is horizontal.

For each ring and ball, we note the temperature at which the bitumen squeezed out by the ball touches the control disk of the apparatus.

4. Penetration according to GOST 11501-73. Essence – measuring the depth of penetration of the penetrometer needle into the test sample at a given load, temperature and time. We place the cup with bitumen in a bath filled with water. The water temperature in the bath should be 25°C. The depth of penetration of the needle is determined at 25°C and 0°C. Remove the cup with the sample from the bath after 60 minutes, and place it in a vessel with water with a capacity of at least 1 liter. We put the container on one hundred of laboratory installation and bring the end of the needle to the surface of the material under study so that the needle touches it slightly. We set the reading to zero, turn on the stopwatch, press the device button, allowing the needle to freely enter

the test sample for 5 seconds. Releasing the button, write down the readings (we take measurements 3 times). The depth of penetration of the needle at 0°C is determined with the changes indicated below.

Pour ice water into the bath. The water temperature should be 0°C, the sample is kept for 60 minutes.

5. Adhesion

Essence – to determine the adhesion of bitumen to a metal surface.

Heat 0.5 g of the test sample on a metal plate. Cover with another hot plate and place a weight of 500 grams on top. We make two samples. When the plates have cooled down, we apply a tensile load and write down the readings in kgf/cm.

6. Heatresistance

The essence is to determine the resistance to heat.

We weigh the plates and pour a certain amount of heated bitumen onto them. After cooling, cut off the edges and weigh the plate with bitumen.

Determine the mass of bitumen before heating (m_{b1}). We install the plate in a vertical position in a muffle furnace and keep it at 125°C for 4 hours. After cooling, cut off the edges of the plate and weigh it. Determine the mass of the remaining bitumen (m_{b2}). Heat resistance is determined by the formula, in%:

$$\% = \frac{m_{б1} - m_{б2}}{m_{б1}} \times 100.$$

RESULTS

When performing the experiment, the optimal synthesis parameters were selected, providing a high yield of the fixer, such as temperature, reaction time, molar ratio of reactants. The results of the experiment are shown in Figures 1–3.

According to the presented graph, as the temperature rises from 30° to 40°C, the yield of the reaction products

Fig. 1. **Influence of the molar ratio of reactantsto the output of dicyandiammonium compound:**
ethylenediamine : propene nitrile : methanal : ethanoic acid
1 – 1,0:2,0:2,1:2,0; 2 – 1,0:2,0:2,0:2,0; 3 – 1,0:2,0:2,0:2,1

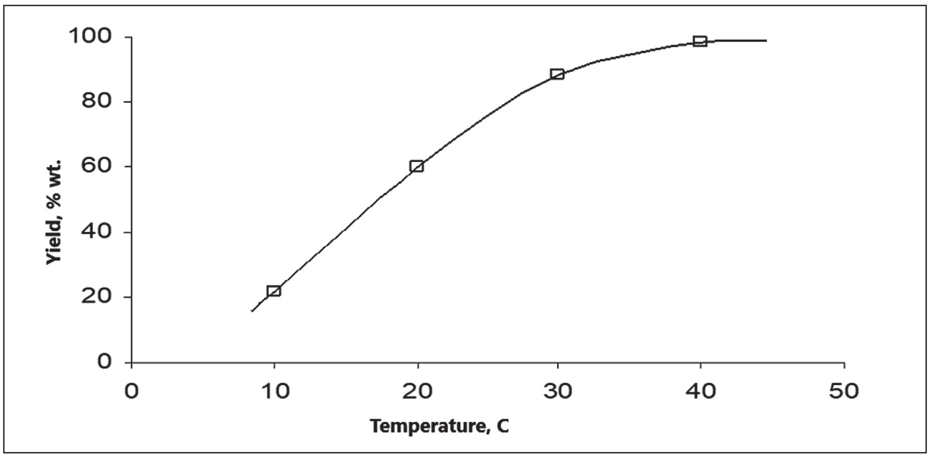

Fig. 2. **Dependence of the yield of dicyandiammonium compounds from temperature**
(* exposure duration 0.5 hours)

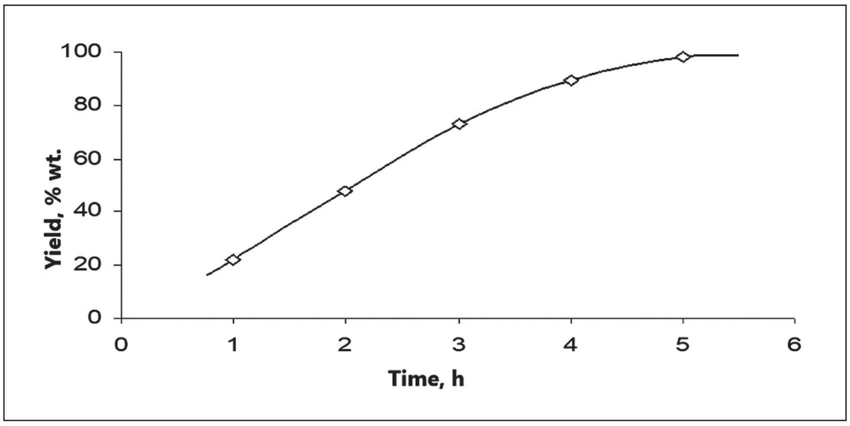

Fig. 3. **Dependence of the yield of dicyandiammonium compounds onreaction time**

increases. A further increase in temperature does not affect the yield of the dicyandiammonium compound.

At a temperature of 40°C, the synthesis of the dicyandiammonium compound is completed in 5.0 h with a yield of the reaction product of 99.0% wt.

The dicyandiammonium compound obtained was identified by the elemental composition of the NMR¹N spectrum.

The obtained NMR¹Nspectra confirm the proposed structure of the synthesized dicyandiammonium compound.

At a temperature of 40°C, the synthesis of the dicyandiammonium compound is completed in 5.0 h with a yield of the reaction product of 99.0% wt.

The obtained dicyandiammonium compound was identified by the elemental composition of the NMR¹N-spectrum, which is shown in Figure 3.13. The NMR¹N spectrum was recorded on a Bruker-AM-300 spectrometer with an operating frequency of 300 MHz, the internal standard is TMS. The spectrum was recorded in deutero-acetone (D-Ac), chemical shifts were measured on the δ-scale and given in ppm, and the spin-spin coupling constant (SSCC) in Hz. NMR¹N spectrum: 1.3 (s, 6H, CH3); 2.35 (m, 4H, CH2); 2.63 (m, 4H, CH2); 2.73 (m, 2H, CH2); 3.05 (m, 2H, CH2); 3.72 (d, 2H, CH2, J = 10); 3.8 (d, 2H, CH2, J = 10).

In the NMR¹N spectrum, due to the presence of symmetry in the molecule, only seven groups of peaks are observed. Intensive multiplet 2.05 ppm. is the resonance signal of the protons of the partially deuterated acetone present in the deuteroacetone. Singlet 1.3 ppm with an integrated intensity of six protons belongs to the methyl protons of two equivalent acetate groups. In the weakest field, the multiplet is 3.72÷3.80 ppm. (integrated intensity – four protons), two equivalent methylene groups with nitrogen atoms and hydroxyl groups in the α-positions resonate. Multiplet 2.35 ppm (integrated intensity – four protons) refers to two methy-

Table 5
Physical and chemical indicators of dicyandiammine

Name	**Indicator**
1. Appearance	Syrupy mass from colorless to yellow
2. Solubility in distilled water at 30°C	complete
3. The relative viscosity of an aqueous solution in a ratio of 1:1 vol. at 20°C, cPs	1.480
4. Density at 20°C, g/cm³	1.0800
5. pH	7.0
6. Acidity, mg KOH	19.1
7. Stability, rpm (3000 rpm, τ = 15 min.)	Resilient (does not delaminate)
8. Mass fraction of nitrogen,% wt.	15.0

lene groups at C ≡ N – group.The other three groups of peaks 2.63 ppm, 2.73 ppm. and 3.05 ppm. belong to four CH_2-groups at nitrogen atoms (the integrated intensity corresponds to eight protons).

The obtained NMR[1]Nspectra confirm the proposed structure of the synthesized dicyandiammonium compound.

The physicochemical parameters of the dicyandiammonium compound are shown in Table 5.

The dicyandiammine synthesized by us has high efficiency when added to bitumen as a fixing agent, is environmentally safe during its synthesis and practical use due to the complete binding of free ethanic acid and methanal in the complex.

DISCUSSION

It follows from the literature review that at present the production of nitrogen-containing compounds is used in many areas of industry.

The current stage of development of the petrochemical industry is currently focused on obtaining products of the main organic and petrochemical synthesis with high technical and operational characteristics.

In this direction, diamines are of scientific and practical interest, as the practical experience of their use confirms, they provide protection of polymer compounds from abrasion, aging, exposure to an aggressive environment, improve their physical and mechanical properties, and have a long service life.

In order to study the effect of the modifier (fixer) synthesized by us on bitumen, the following methods of analysis were carried out: penetration, softening temperature (ring-and-ball method).

An increase in the concentration of the fixer in the test samples did not lead to its stratification with bitumen, which indicates their compatibility. The experimental results are shown in table 6.

The presented data indicate an increase in the penetration index with an increase in the mass fraction of dicyandiamine. That is, an excessive increase in the content of dicyandiamine leads to a decrease in the hardness of the samples, which indicates a decrease in the value of such an indicator as surface tension.

Measurements of the softening point (using the ring-and-ball method) showed that with an increase in the content of the fixer, an increase in the softening temperature occurs, which means that the modified samples

Table 6
Dependence of indicators on the concentration of the modifier

Nameofsamples	Qualitativeindicators	
	Penetration, mm	Temperature softening (Ts), °C
Initialbitumen	3.1	95
Bitumen with 10% additive	3.3	98
Bitumen with 20% additive	3.6	100
Bitumen with 30% additive	3.9	102
Bitumen with 40% additive	4	103

Table 7
Research results at various concentrations

Concentration, %	Softeningpoint, °C	Penetrationat 25°C, mm	Penetrationat 0°C, mm	Adhesion, kgf/cm	Heatresistance, %
10	160	3.3	6	41	16.7
20	163	3.6	6	39	16.0
30	165	3.9	6.3	35	15.7
40	167	4	6.5	32	15.0
50	168	Not a homogeneousmixture			
60	169	Not a homogeneousmixture			

are not subject to softening at the temperature at which the initial sample softens and softens only with a further increase in temperature, and with an increase in the content of the fixer, the difference is Ts only increases.

Thus, the proposed bitumen fixer, at various ratios, gives the product elasticity and increased resistance to softening with increasing temperature.

In order to study the operational characteristics of road bitumen for its compatibility with the fixer, experiments were carried out at higher softening temperatures.

Compounding was carried out at a temperature of 210°C for 60 minutes. The resulting products have been tested to determine their physical and mechanical characteristics. The research results are presented in table 7.

According to the presented data in the table, the best properties of bitumen with a modifier were found at a concentration of a fixer of 30–40% by weight.

It can also be noted that an increase in temperature contributed to an improvement in technical performance. Characteristics of the obtained samples: homogeneous structure, high softening point and good adhesion properties. It was also observed that an increase in the content of the fixer leads to the destabilization of the dispersed structure of bitumen.

CONCLUSION

Thus, the outcome of the study is a fixative based on an additive, a derivative of triethylenediaminedicyan, which led to the formation of a nanodispersed structure of bitumen of the "sol-gel" type, the quality indicators of which would meet the requirements of the new standard GOST 33133-2014 "Viscous road oil bitumen". Its synthesis laws and physical and chemical parameters were studied, the structure was proved.

The proposed bitumen fixer, at various ratios, gives the product elasticity and increased resistance to softening with increasing temperature.

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