

Limited Liability Company
“FTS”

PROPRIETARY STANDARD

**“GRANULIT” STABILIZING AGENT FOR BITUMINOUS STONE MASTIC
MIXTURES**

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Introduction

The objectives and principles of standardization in the Russian Federation are set forth in the Federal Law No.184-FZ “On Technical Regulation” dated 27th December 2002, while the rules of application of proprietary standards are specified in GOST R 1.4-2004 “Standardization in the Russian Federation. Proprietary Standards. Basic Provisions”.

Information about the Standard

1. The Standard was DEVELOPED by Limited Liability Company “FTS”.
2. The Standard was INTRODUCED by Limited Liability Company “FTS”.
3. The Standard was APPROVED AND BROUGHT INTO EFFECT by the Order No.187 of General Director of “FTS” LLC dated 17th March 2015.
4. This Standard implements provisions of Articles 11 to 13, and 17 of the Federal Law “On Technical Regulation”.
5. This Standard was developed taking into consideration recommendations provided in ODM 218.1.001-2010 and ODM 218.1.002-2010.
6. This Standard is INTRODUCED FOR THE FIRST TIME.

“RIMINVEST” LLC located in Nizhny Novgorod is the official distributor of “GRANULIT” products on the territory of Russia and CIS countries; Tel.: 8(831) 423-55-82, 228-16-17; Website: www.riminvest.ru

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PROPRIETARY STANDARD

“GRANULIT” STABILIZING AGENT FOR BITUMINOUS STONE MASTIC MIXTURES**Technical Specifications**

Introduced on March 17, 2015

1. Scope of Application

This standard covers “GRANULIT” stabilizing agent for bituminous stone mastic mixtures (hereafter referred to as the “Stabilizing Agent”) made of cellulose fibers. This Stabilizing Agent is intended for sorption (retention) of binding agents in bituminous stone mastic mixtures in the process of preparation of the mixtures, their transportation, and paving.

2. References to Applicable Regulations

This document contains references to the following standards:

GOST R 12.4.230.1-2007 System of Occupational Safety Standards. Protective Goggles. Basic Technical Requirements;

GOST 12.1.004-91 System of Occupational Safety Standards. Fire Safety. Basic Requirements;

GOST 12.1.005-88 System of Occupational Safety Standards. Basic Sanitary and Hygienic Requirements to Air in Working Zones;

GOST 12.1.004-89 System of Occupational Safety. Fire and Explosion Hazards of Substances and Materials. Nomenclature of Parameters and Methods of Their Estimation;

GOST 12.4.011-89 System of Occupational Safety Standards. Personal Protective Equipment. Basic Requirements and Classification;

GOST 12.4.028-76 System of Occupational Safety Standards. “Petal” IIIБ-1 Breathing Masks. Technical Specifications;

GOST 12.4.103-83 System of Occupational Safety Standards. Purpose-Specific Protective Clothes and Individual Protection Equipment for Feet and Hands. Classification;

GOST 12.4.021-75 System of Occupational Safety Standards. Ventilation Systems. Basic Requirements;

GOST 6617-76 Construction Petroleum Bitumen. Technical Specifications;

GOST 8269.0-97 Crushed Stone and Gravel from Dense Rock, and Waste Products for Construction Works. Physical and Mechanical Test Methods Specifications;

GOST 10700-97 Waste Paper and Waste Cardboard. Technical Specifications;

GOST 14192-96 Cargo Marking;

GOST 22245-90 Viscous Road Petroleum Bitumen. Technical Specifications;

GOST 27574-87 Women’s Clothes for Protection against General Industrial Contamination and Mechanical Factors;

GOST 27575-87 Men’s Clothes for Protection against General Industrial Contamination and Mechanical Factors;

GOST 30108-94 Construction Materials and Products. Determination of Specific Effective Activity of Natural Radionuclides; and

GOST 31015-2002 Bituminous Stone Mastic Mixtures and Stone Mastic Asphalt. Technical Specifications.

Note: When using this standard, please, verify legal effect of the standards (and classifiers) to which references are made herein under respective indexes of standards (and classifiers) valid as of 1st January of the current year and respective reference indexes published in the current year. If a document to which references are made has been superseded (amended), you should be guided by the superseding (amended) standard when using this standard. If a document to which references are made has been cancelled and not replaced, provisions in which references are made to the said document will apply in part not involving the reference.

3. Terms, Definitions, and Abbreviations

The following terms with respective definitions are used in this standard:

3.1. Bituminous stone mastic mixture is a rationally selected mixture of mineral materials (crushed stone, crushed sand, and mineral powder), road bitumen (with polymer or any other additive, or without additives), and a stabilizing agent taken in specific proportions and mixed in a heated state.

3.2. Stone mastic asphalt is a compacted bituminous stone mastic mixture.

3.3. Stabilizing agent is a substance having stabilizing effect on bituminous stone mastic mixtures and ensuring its resistance to segregation.

4. Classification

The Stabilizing Agent comes in two commercial types (grades):

Granulit is a stabilizing agent in the form of brown granules 7 to 10 mm long and 5 mm in diameter, which is made of cellulose fibers. These granules are treated with a stabilizing substance to ensure binding. The Stabilizing agent is not hygroscopic; it is resistant to atmospheric humidity and does not cake in the process of storage.

Granulit-66 is a stabilizing agent in the form of dark-brown granules up to 10 mm long and 4 mm in diameter, which is made of cellulose fibers treated with modified bitumen. This material is characterized by high density of its granules and low absorbability, which ensure convenience of transportation of the Stabilizing Agent and its further storage at production sites.

5. Technical Requirements

5.1. Basic Parameters and Characteristics

5.1.1. The Stabilizing Agent shall meet all requirements set forth in this standard and be manufactured under process control documentation approved following the established procedure.

5.1.2. The Stabilizing Agent is made of cellulose fibers.

5.1.3. The cellulose fibers shall have ribbon structure with fibers 0.1 to 2.0 mm long. The fibers shall be homogeneous and free of bundles, clusters of non-crushed materials, and foreign matters.

5.1.4. By its physical and mechanical properties the Stabilizing Agent shall meet the requirements set forth in Table 1.

Table 1 – Physical and Mechanical Properties of “Granulit” Stabilizing Agents

No.	Parameter	Value	
		Granulit	Granulit-66
1	Appearance and colour	Cylindrical brown granules up to 10 mm long and 5-6 mm in diameter	Cylindrical dark-brown granules up to 10 mm long and 4 mm in diameter
2	Humidity, mass %	3 - 7	3 - 7
3	Bulk density, kg/m ³	450 - 500	500 - 650

4	Heat resistance at 220°C by changes in its mass in the process of heating, mass %	Not exceeding 7.0	Not exceeding 7.0
5	Binder outflow rate, Ep, mass %	Not exceeding 0.2	Not exceeding 0.2
6	Content of fibers 0.1 to 2.0 mm long, %, at least	80	80
<p>Note: By the binder outflow rate (Ep) depending on the grade, composition, and quality of materials used for bituminous stone mastic mixtures, the amount of “Granulite” stabilizer added may vary from 0.3 to 0.6%.</p>			

5.2. Requirements to Materials

The Stabilizing Agent shall be made of the following materials:

- Recycled waste paper of group A or B under GOST 10700;
- Cellulose, chemical, or organic fibers under regulatory and technical documents applicable to a specific type of fibers;
- Binding materials such as petroleum bitumen under GOST 22245 and GOST 6617, modified polymer-bitumen binders under GOST R 52056, and other improved bitumen binders under regulatory and technical documents which have been agreed with and approved by the Customer following the established procedure; and
- Thermostabilizing agents under regulatory and technical documents.

5.3. Marking

5.3.1. The Stabilizing Agent shall be marked in accordance with requirements set forth in GOST 14192.

5.3.2. Each pack unit should be marked. The marking shall be clear and made using indelible paints.

5.3.3. Each pack unit shall be marked as follows:

- Country of origin;
- Manufacturer's name and (or) trade mark and address;
- Batch and lot number and date of manufacture;
- Net mass, kg;
- Warranty storage period; and
- Reference designation of this standard.

5.4. Packaging

5.4.1. The Stabilizing Agent is packed in leak-proof packages ensuring preservation of the mass and properties of the Stabilizing Agent in the process of transportation and storage, i.e. in bags of capacity of more than 1 m³ (big bags), and polyethylene packages. Mass of the Stabilizing Agent in a package shall not exceed 8 kg.

5.4.2. The package shall protect the Stabilizing Agent against penetration of moisture from the environment.

5.4.3. The pack unit mass shall be approved by the end user.

6. Safety Requirements and Environmental Protection

6.1. Raw materials shall be used in the manufacturing process so as to meet all safety requirements set forth in relevant regulatory documents.

6.2. Persons involved in the process of manufacture of the Stabilizing Agent shall undergo primary (at their admission to work) and regular medical checkups and use individual protective equipment under GOST 12.4.011, GOST 12.4.028, and GOST 12.4.103.

6.3. Hazardous substance content in air in working zones shall not exceed maximum permissible levels specified in GOST 12.1.005. The production facilities shall be equipped with general, draw-in, and exhaust ventilation systems under GOST 12.4.021 which will ensure conditions in working zones complying with requirements set out in GN 2.2.5.1313 “Maximum Permissible Concentrations (MPC) of Hazardous Substances in Air in Working Zones” [1]. The manufacturing process shall meet requirements set forth in SN 2.2.1327 “Hygienic Requirements to Organization of Manufacturing Processes, and Arrangement of Production Equipment and Tools” [2].

6.4. Emission of hazardous chemical substances by the Stabilizing Agent shall not exceed maximum permissible concentrations (MPC) approved by healthcare authorities.

6.5. Persons working with the Stabilizing Agent shall be equipped with:

- Eye protection equipment such as protective goggles under GOST R 12.4.230.1; and
- Protective clothes under GOST 27574 and GOST 27575.

Only persons who have received instructions on occupational safety shall be admitted to works.

6.6. All persons involved in the manufacturing process shall be trained to provide first aid.

First aid kits complete with drugs required for provision of first pre-hospital aid shall be available in all production facilities.

6.7. The Stabilizing Agent shall be manufactured, used, stored, and tested in observance of requirements to fire safety under GOST 12.1.004.

6.8. In order to avoid pollution of the environment, all components of the Stabilizing Agent shall be stored as follows:

- Bitumen and superficially active substances shall be stored in tightly closed containers; and
- Recycled water paper, cellulose, and chemical and organic fibers shall be stored in tightly closed containers.

6.9. Specific effective activity (A_{eff}) of natural radionuclides in the Stabilizing Agent shall not exceed 740 Bq/kg.

7. Acceptance Procedure

7.1. The Stabilizing Agent shall be accepted in batches. A batch is a quantity of the stabilizing agent of the same quality, which is made of the same raw materials following the same manufacturing procedure.

7.2. The manufacturer shall enclose a document confirming quality of the Stabilizing Agent to each batch or its part (if a batch will be distributed between different facilities), which shall contain the following:

- Manufacturer's name and (or) trade mark;
- Name of the Stabilizing Agent;
- Batch and lot number and date of manufacture;
- Net mass;
- Type of containers or packages and number of pack units in this lot;
- Warranty storage period; and
- Results of acceptance tests.

7.3. In order to verify whether the Stabilizing Agent complies with the requirements set forth in this standard, the manufacturer shall perform acceptance and periodic tests.

7.4. To verify compliance of the Stabilizing Agent with this standard, spot samples are taken of 10% of pack units of each batch (at least three pack units) at least 100 mm from the surface of the material. If mass of a pack unit exceeds 100 kg, spot samples are taken at three levels of the entire volume of the unit:

– Upper level (up to 200 mm from the surface); – Middle level (from the middle part of a pack unit); – Lower level (up to 200 mm from the bottom of a pack unit).

Spot sampling is performed manually or using samplers. A spot sample shall be at least 0.5 kg.

After sampling the spot samples are joined together, mixed up thoroughly, and reduced using the quantization method. To quantize a sample, the material is leveled and divided into four parts by perpendicular lines going through the center. Any two opposite quarters are sampled into a lab sample. A sample is reduced two times, four times, etc. by sequential quantization, until a mass suitable for all tests provided for in this standard is reached.

A sampling report should be executed for each lab sample, which shall specify the Stabilizing Agent, manufacturer's name, address of its production facilities, sampling place and date, mass of the lab sample, and positions, full names and signatures of persons performing sampling.

A lab sample shall be packed and stored in a leak-proof container so as to ensure preservation of the mass and properties of the Stabilizing Agent till the moment of testing. Each sample shall be marked with two labels with reference designation of the sample (one of these labels shall be put into the package, the other one shall be stuck to the outer packaging). For each test an analytical sample is taken from each lab sample using the quantization method. Some quantity of such analytical sample is weighed for analysis in accordance with the test methods used.

7.5. Each batch of the Stabilizing Agent is subject to acceptance tests in the process of which the following parameters are verified:

- Appearance and colour;
- Moisture content;
- Bulk density of granules; and
- Presence of marking and integrity of packages.

7.6. In the process of periodic tests the following parameters are verified:

- Heat resistance at 220°C by changes in mass of the Stabilizing Agent in the process of heating; and
- Binder outflow rate, Ep.

Such periodic tests shall be performed at least once in six months.

7.7. At least once a year and at each replacement of suppliers of materials the manufacturer shall perform periodic tests to estimate the specific effective activity (A_{eff}) of natural radionuclides.

7.8. Parameters of fire and explosion hazards of materials used in the process of manufacture of the Stabilizing Agent shall be determined when the Stabilizing Agent is launched into manufacture, and if suppliers are replaced.

7.9. The end user is entitled to check if the Stabilizing Agent delivered meets all requirements set forth in this standard using the sampling and testing methods provided for herein.

7.10. If results of testing are unsatisfactory by at least one of the parameters given above, the Stabilizing Agent shall be re-tested by this unsatisfactory parameter. Results of such re-testing shall apply to the entire batch and deemed final.

If results of such re-testing are unsatisfactory, this batch of the Stabilizing Agent shall not be accepted.

8. Control Methods

8.1. Raw materials used in the process of manufacturing of the Stabilizing Agent shall be tested pursuant to requirements set forth in relevant regulatory or technical documents.

8.2. The appearance and colour shall be assessed visually to verify their compliance with the requirements set forth herein.

8.3. The binder outflow rate shall be estimated under Appendix A.

8.4. The bulk density of cellulose fibers shall be estimated under Appendix B.

8.5. The moisture content and heat resistance of fibers shall be estimated under Appendix C.

8.6. The specific effective activity (A_{eff}) of natural radionuclides in raw materials used to manufacture the Stabilizing Agent or in the Stabilizing Agent directly shall be estimated under GOST 30108.

9. Transportation and Storage

9.1. The Stabilizing Agent shall be transported by all types of transport in packaging containers provided by the manufacturer. In the process of transportation all transport regulations shall be abided by.

9.2. The Stabilizing Agent shall be stored in the original packaging in indoor warehouses. In the process of storage all current fire safety regulations shall be abided by.

9.3. In the process of transportation and storage all measures shall be taken to protect the Stabilizing Agent against wetting and contamination and ensure integrity of its packaging.

10. Instructions for Use

10.1. Standard consumption rates are estimated on the basis of results of laboratory testing of bituminous stone mastic mixtures. The standard rate of consumption of the Stabilizing Agent to be added to mixtures, which is recommended by its manufacturer, is 0.2 to 0.6% in excess of the mineral component.

10.2. The Stabilizing Agent shall be loaded to the mixer of an asphalt-mixing plant using purpose-specific dosing devices or any other volume-weighing methods.

10.3. To evenly distribute the Stabilizing Agent in asphalt concrete mixtures, the Stabilizing Agent shall be pre-mixed with a hot mineral material and then with bitumen. Duration of mixing shall be determined on the basis of technical parameters of the mixer.

11. Manufacturer's Warranties

11.1. The manufacturer warrants that quality of the Stabilizing Agent meets all requirements set forth in this standard provided that the end user abides by all established rules and procedures of transportation, storage, and use.

11.2. The warranty storage period is 12 months from the date of manufacture.

11.3. Upon expiration of the warranty period the Stabilizing Agent may be used as intended, if compliance of its quality with the requirements of this standard has been verified.

Appendix A (mandatory)

Method of Evaluation of Resistance to Segregation of a Mixture by its Binder Outflow Rate

This method is developed to evaluate ability of a hot bituminous stone mastic mixture to retain binders it contains.

A.1. Control measuring devices and auxiliary equipment:

- Analytical laboratory balance, accuracy class 4 under GOST 24104;
- 1000 cm³ heat-resistant beakers 10 cm in diameter under GOST 23932;
- Cover-glasses;
- Mercury-in-glass chemical thermometer with the measurement range from 100°C to 200°C with a smallest scale division value not exceeding 1°C; and
- Drying cabinet.

A.2. Test preparation procedure:

A prepared bituminous stone mastic mixture is heated to the maximum temperature given in Table 3 and mixed up thoroughly. A drying cabinet is also heated to the said temperature which will be maintained during the test with a tolerated error $\pm 2^\circ\text{C}$.

An empty beaker is weighed, put into the drying cabinet, and held at the temperature given in Table 3 for at least 10 min. The beaker is then put onto an analytical balance. Some mixture (0.9 to 1.2 kg) should be promptly put into the beaker which is then weighed and closed by a cover-glass.

A.3. Test Procedure

The beaker containing the mixture is put into the drying cabinet where it is held at the maximum temperature given in Table 3 for (60 ± 1) minutes. The beaker is then taken out and uncovered. The mixture is removed from the beaker by turning the beaker upside down and slightly shaking it for (10 ± 1) s. The beaker is then put onto a table with its bottom down, and particles of the mixture (more than 2 mm in size) sticking to the bottom or sides of the beaker are removed using a forceps. The beaker is left to cool for 10 min and then weighed together with remaining binder sticking to its internal surface.

A.4. Test results processing:

Binding outflow in mass % is calculated using the following formula:

$$B = \frac{g_3 - g_1}{g_2 - g_1} \times 100 \quad ,$$

where g_1 is the mass of the empty beaker, g;
 g_2 is the mass of the beaker containing the mixture, g; and
 g_3 is the mass of the beaker after removal of the mixture, g.

The test result is calculated as the mean arithmetic of two parallel calculations rounded off to two decimal places. A discrepancy between results of two tests performed in parallel shall not exceed 0.05% of the absolute value. If the discrepancy is big, the mixture should be re-tested for evaluation of binder outflow. In this case, findings of all four evaluations are taken into calculation of the mean value.

Appendix B (mandatory)

Evaluation of Solid Density of the Stabilizing Agent

This method is developed to evaluate density of a stabilizing agent without its pores.

B.1. Control measuring devices and auxiliary equipment:

- 250 ml measuring flasks under GOST 1770;
- Analytical balance, accuracy class 4 under GOST R 53228;
- A sand bath or a closed-coil heating plate; and
- Distillated water under GOST 6709.

B.2. Test preparation procedure:

Two weighed portions (for two parallel evaluations) of about 5 mg are taken from a stabilizing agent sample taken for composition adjustment of stone mastic asphalt. Each portion of the stabilizing agent is placed into a clean, dry and weighed measuring flask which is then weighed and filled with distilled water by 1/3. If granulated hydrophobic stabilizers are tested, a wetting agent shall be added to the water. Such wetting agents include powder, paste, or liquid technical or household detergents. The wetting agent is added to water in the following proportions per liter of water: 15 g of a liquid detergent; 10 g of a paste detergent (dissolved in water in proportion 1:1); and 3 g of a powder detergent. Solid density of such wetting agent solutions is calculated using the bottle method under GOST 3900.

B.3. Testing procedure:

Contents of the flask are stirred and boiled on a sand bath during 1 h, and then left to cool down to ambient temperature. The flask is then filled with the said wetting agent solution to the level marked of the flask neck. The flask is weighed. After that all the content is removed from the flask which is then rinsed and filled with the wetting agent solution of ambient temperature to the level marked on its neck, and weighed again.

B.4. Test results processing:

Solid density of the stabilizing agent, ρ , g/cm³, is calculated using the following formula:

$$\rho = \frac{(m - m_1)\rho_c}{m - m_1 + m_2 - m_3},$$

where m is the mass of the flask containing the stabilizing agent, g;

m_1 is the mass of the empty flask, g;

m_2 is the mass of the flask with the wetting agent solution, g;

m_3 is the mass of the flask containing the stabilizing agent with wetting agent solution, g;

ρ_c stands for density of the wetting agent solution, g/cm³.

The result of each test is rounded off to two decimal places. An absolute tolerated discrepancy between results of two tests performed in parallel shall not exceed 0.02 g/cm². If the absolute tolerated discrepancy is exceeded, the mixture should be re-tested till a tolerated discrepancy is reached. Solid density is calculated as the mean arithmetic of results of two parallel tests.

Appendix C **(mandatory)**

Estimation of Moisture Content and Heat Resistance of Fibers

This method is developed to evaluate mass loss of fibers at a pre-set temperature and test duration.

C.1. Control measuring devices and auxiliary equipment:

Rectangular metal trays 20 x 10 x 2 cm in size;

Drying cabinet with a temperature controller maintaining the pre-set temperature accurate to $\pm 3^{\circ}\text{C}$;

Mercury-in-glass thermometer with a smallest scale division value not exceeding 1°C ;

Desiccator with anhydrous calcium chloride under GOST 23932; and

Analytical laboratory balance, accuracy class 4 under GOST 24104.

C.2. Test preparation procedure:

Before testing, a sample of fibers is placed on a sheet of paper and loosened manually to remove small lumps if these are present in the sample.

Metal trays which have been washed thoroughly are put into the drying cabinet heated to $(105 \pm 3)^{\circ}\text{C}$ for at least 30 min, and then left in a desiccator to cool down to ambient temperature.

C.3. Testing procedure:

In the process of testing the fibers are weighed with a tolerated weighing error of 0.1% of the fibers mass. The mass is measured in grams rounded off to two decimal places.

This test is performed on two trays. Each tray prepared as provided in Item C.2 is weighed. Two weighed portions of (5 ± 1) g are taken from a fiber sample prepared as provided in Item C.2 and put onto the trays which should be filled evenly but loosely. The trays filled with fibers are weighed and put into the drying cabinet heated to a temperature of $(105 \pm 3)^{\circ}\text{C}$ to dry the fibers.

In 30 minutes the trays with fibers are taken out of the drying cabinet, placed into the desiccator and left to cool down to ambient temperature. After that the trays are weighed and placed into the desiccator again.

The trays filled with fibers dried in the drying cabinet at a temperature of $(105 \pm 3)^{\circ}\text{C}$ and cooled in the desiccator to ambient temperature are put into the drying cabinet heated up to $(220 \pm 3)^{\circ}\text{C}$.

Temperature is adjusted using a thermometer the mercury-filled reservoir of which is positioned at the level of the trays.

Since the temperature in the drying cabinet drops when cold trays are put into it, the time during which the trays with fibers are kept in the drying cabinet should be counted from the moment at which the pre-set temperature is reached.

The trays with fibers are kept in the drying cabinet for 5 min at a temperature of $(220 \pm 3)^\circ\text{C}$.

After that the trays with fibers are taken out of the drying cabinet, placed into the desiccator, left to cool down to ambient temperature, and weighed.

C.4. Test results processing:

Moisture content in fibers, W , %, is calculated using the following formula:

$$W = \frac{g_2 - g_3}{g_3 - g_1} \times 100,$$

where

g_1 is the mass of the tray, g;

g_2 is the mass of the tray filled with the fibers, g; and

g_3 is the mass of the tray with the fibers after drying in the drying cabinet, g.

Heat resistance of fibers, T_f , %, is calculated using the following formula:

$$T_f = \frac{g_3 - g_4}{g_3 - g_1} \times 100,$$

where

g_4 is the mass of the tray filled with fibers after drying in the drying cabinet at a temperature of $(220 \pm 3)^\circ\text{C}$, g.

The discrepancy between results of two tests performed in parallel shall not exceed 0.5% (of the absolute value). The result of this test is the mean arithmetic value of two tests performed in parallel, which is rounded off to one decimal place.

References

- [1] GN 2.2.5.1313-03 Maximum Permissible Concentrations (MPC) of Hazardous Substances in Air in Working Zones
- [2] SN 2.2.2.1327-03 Hygienic Requirements to Organization of Manufacturing Processes, and Arrangement of Production Equipment and Tools

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