

**POLYMETHYLSILOXANE FLUID
OF GRADE PMS-200 (ПМС-200)
SPECIFICATIONS
GOST 13032-77
(Abstract)**

The standard refers to polymethylsiloxane fluid of grade PMS-200 (IIMC-200) which is a mixture of polymers of linear and branched structure.

1 Technical Requirements

Polymethylsiloxane fluid of grade PMS-200 (IIMC-200) shall be produced as per the present standard based on the process regulations approved in accordance with the established procedure.

As regards physical and chemical parameters, fluid PMS-200 (IIMC-200) shall comply with the standard values and requirements given in Table 1.

Table 1

Parameter	Standard value	Test method
1 Exterior view	Transparent colorless liquid	As per item 3.2
2 The content of mechanical impurities	None	As per item 3.2
3 Viscosity-to-density ratio: at 20 °C, m ² /s (cSt)	(192–208) · 10 ⁻⁶ 192–208	As per item 3.3
4 Dynamic viscosity at minus 50 °C, Pa · s (cP), maximum	2.0 (2 000)	As per item 3.4
5 Flash point in the open crucible, °C, minimum	316	As per item 3.5
6 Setting point, °C, maximum	Minus 62	As per item 3.6
7 Reaction of medium (pH of aqueous extract)	6.2–7.0	As per item 3.7
8 Weight ratio of silicium, %	37.0–38.5	As per item 3.8
9 Water content, mass %, maximum	0.004	As per item 3.9

2 Acceptance Rules

2.1 The fluid shall be accepted in batches. A batch is any amount of fluid of uniform quality accompanied by one quality document.

The document shall give the following information:

- Manufacturer's name and trademark;
- Product name;
- Manufacture date;
- Batch number;
- Gross weight and net weight;
- Results of analysis or confirmation of product quality compliance with the requirements of the present standard;
- Designation of the present standard.

2.2 To control fluid quality, 10 % of production units shall be taken from a batch, but no less than three production units in case of batches consisting of 30 production units or less.

2.3 In the event of unacceptable analysis results obtained by at least one parameter, a repeated analysis of a duplicate test sample taken from the same batch shall be carried out.

The results of the repeated analysis apply to the whole batch.

3 Analysis Methods

3.1 Sampling Methods

3.1.1 Before sampling, the fluid shall be mixed thoroughly. Snap samples are taken using a dry clean glass dropping tube, dipping to container bottom.

3.1.2 Snap samples are joined together, mixed thoroughly, and an average sample of 0.5 dm³ is taken from the obtained sample; the average sample is placed into a clean dry jar with a ground plug. A label shall be attached to the jar, specifying: product name, batch number, date of sampling.

3.1.3 The average sample shall be mixed before each analysis.

3.2 Determination of Exterior View and Content of Mechanical Impurities

The exterior view of polymethylsiloxane fluid of grade PMS-200 (ПМС-200) and the content of mechanical impurities in it shall be determined visually inside a cylinder of dia. 25–30 mm, made of colorless transparent glass in transmitted light.

3.3 Viscosity-to-Density Ratio Determination

The principle of the method consists in measuring effusion time of a certain amount of the tested fluid under the force of gravity.

3.3.1 Equipment, Materials and Reagents Applied

3.3.1.1 Capillary viscometers made of glass with low thermal-expansion coefficient (e.g. borosilicate glass).

It is allowed to use automatic viscometers.

Viscometers of types VPZhT-1 (ВПЖТ-1), VPZhT-2 (ВПЖТ-2), VPZhT-4 (ВПЖТ-4), VNZhT (ВНЖТ). It is allowed to use viscometers of types VPZh-1 (ВПЖ-1), VPZh-2 (ВПЖ-2), VPZh-4 (ВПЖ-4), VNZh (ВНЖ).

A set of viscometers shall be available for each viscosity range. To make it not necessary to introduce a kinetic energy correction, all these viscometers are designed for the minimum effusion time of 200 s.

Viscometers of different types are given in Figure 1.

3.3.1.2 Supports or other fastening devices for viscometers. To verify vertical position, a plumb line is used.

3.3.1.3 Thermostatic oven or viscometer bath. To fill the bath, it is necessary to use a transparent liquid remaining in the liquid state at the measurement temperature. The bath to be used can be any transparent vessel of such depth that the product placed into viscometer is immersed to at least 20 mm below liquid level in the bath and remains at least 20 mm above the bottom of the bath. The bath shall be equipped with a device enabling to precisely adjust liquid temperature in the bath. The maximum variation of liquid temperature along the length of viscometers and between the installation point of individual viscometers and thermometer installation point shall not exceed:

± 0.01 °C – at temperatures from plus 15 to plus 100 °C;

± 0.03 °C – at temperatures outside of this range.

To fill the thermostatic oven, use is made of:

Industrial ethyl alcohol or synthetic industrial ethyl alcohol, or industrial isooctane – for temperatures from minus 60 to plus 15 °C;

Distilled water – for temperatures from plus 15 to plus 60 °C.

To cool down the liquid in the thermostatic oven, use is made of ice, carbon-dioxide ice (dry ice) or liquid nitrogen.

3.3.1.4 Thermometers

Thermometers with characteristics given in Table 2, correctable to an accuracy of ± 0.02 °C, providing for a possibility to measure with an accuracy of 0.01 °C, or other devices for measuring temperature with similar accuracy.

Table 2

Thermometer measurement ranges, °C	Test temperature, °C	Division value, °C
From minus 51.6 to minus 34	From minus 51 to minus 35	0.1
From minus 19.2 to minus 16.4	minus 17.8	0.05
From plus 23.6 to plus 26.4	plus 25	0.05
From plus 38.6 to plus 41.4	plus 40	0.05
From plus 58 to plus 62	plus 60	0.05

3.3.1.5 Stopwatches or other instruments ensuring time reading with an accuracy up to 0.2 s and an error of $\pm 0.07\%$.

Time reading can be done using electrical instruments, provided current frequency regulation with an error not greater than 0.05 % is ensured.

3.3.1.6 Drying oven, providing heating temperature from 100 to 200 °C.

3.3.1.7 Sieve with mesh size 75 μm , filtering funnels or crucibles.

3.3.1.8 Laboratory filter paper.

3.3.1.9 Coarse-crystalline sodium chloride or anhydrous sodium sulfate.

3.3.1.10 Nefras petroleum solvents S₂-80/120 (C₂-80/120) and S₃-80/120 (C₃-80/120).

3.3.1.11 Nefras-S 50/170 (Нефрас-С 50/170).

3.3.1.12 Petroleum-ether.

3.3.1.13 Acetone.

3.3.1.14 Toluene.

3.3.1.15 High-purity industrial rectified ethyl alcohol.

3.3.1.15 Chromic acid mixture.

3.3.1.16 Hydrochloric acid

3.3.1.17 Distilled water

3.3.2 Test Preparation

3.3.2.1 The test is performed using a clean dry viscometer with measurement range corresponding to the expected viscosity of a test specimen.

3.3.2.2 The temperature of thermostating liquid along viscometer length shall comply with the requirements of item 3.3.1.3.

When measuring temperature in the thermostatic oven, a correction to thermometer reading for the outstanding column of mercury or spirit in the thermometer shall be made.

Calculate correction for the outstanding column of mercury or spirit Δt , °C, using the following formula:

$$\Delta t = K \cdot h \cdot (t_1 - t_2),$$

where K – correction factor, which is equal to 0.00016 for a mercury-filled thermometer and

0.001 for a spirit thermometer;

h – height of the outstanding column of mercury or spirit expressed in degree divisions of the thermometer scale;

t_1 – specified temperature in the thermostatic oven, °C;

t_2 – ambient temperature near the middle of the outstanding column of mercury or spirit, °C (determined with the help of an auxiliary thermometer whose bulb is positioned at the middle of the outstanding column height).

A positive correction is extracted from a given temperature of viscosity determination, and a negative one is added to it, and this temperature is maintained during the tests.

3.3.2.3 Between consecutive measurements, viscometer shall be several times washed with a solvent completely miscible with the tested product, followed by flushing with a completely vaporizable solvent. The viscometer shall be dried in a drying oven at a temperature within plus 100 to plus 120 °C or by passing through it a light flow of clean dry air until complete removal of solvent traces.

The viscometer shall be regularly washed with chromic acid mixture so as to remove organic deposits, thoroughly flushed with distilled water and acetone in succession, and dried with clean dry air. If deposits of inorganic salts are present, before washing with chromic acid mixture they shall be removed using hydrochloric acid, especially when presence of barium salts is suspected.

3.3.2.4 The sample taken is filtered through a sieve, glass or paper filter.

If water is present in the polymethylsiloxane fluid, it shall be dried out using anhydrous sodium sulfate or incinerated coarse-crystalline sodium chloride (NaCl) and filtered through a paper filter.

To obtain homogeneous results of viscosity determination at temperatures below 95 °C, the product shall be preheated.

For that purpose, heat up the tested product in its container to approximately 50 °C, while stirring and shaking it. Check the bottom of the container with a rod to make certain that all paraffinic substances have been dissolved. Pour 100 cm³ of the tested product into a conical flask. Plug the flask loosely with a cork or rubber stopper. Dip the flask into a bath with boiling water for 30 min. Mix thoroughly, take the flask out of the bath, and filter the sample in a drying oven; keeping the temperature steady, fill a viscometer heated up to the test temperature and place it into thermostatic oven. Viscosity shall be determined not later than within 1 hour from heating.

3.3.2.5 At test temperatures below the dew point, tubes loosely filled with desiccating agent shall be put on the exposed viscometer joints, so as to prevent water condensation. The tubes shall not prevent the tested product from effusing under the action of changing pressure in the viscometer.

At test temperatures below 0 °C viscometer is filled with the tested product at the ambient temperature and then cooled down to the bath temperature, while keeping tested product in the capillary.

3.3.3 Test Procedure

3.3.3.1 Viscometer (Figure 1) is filled with the tested product.

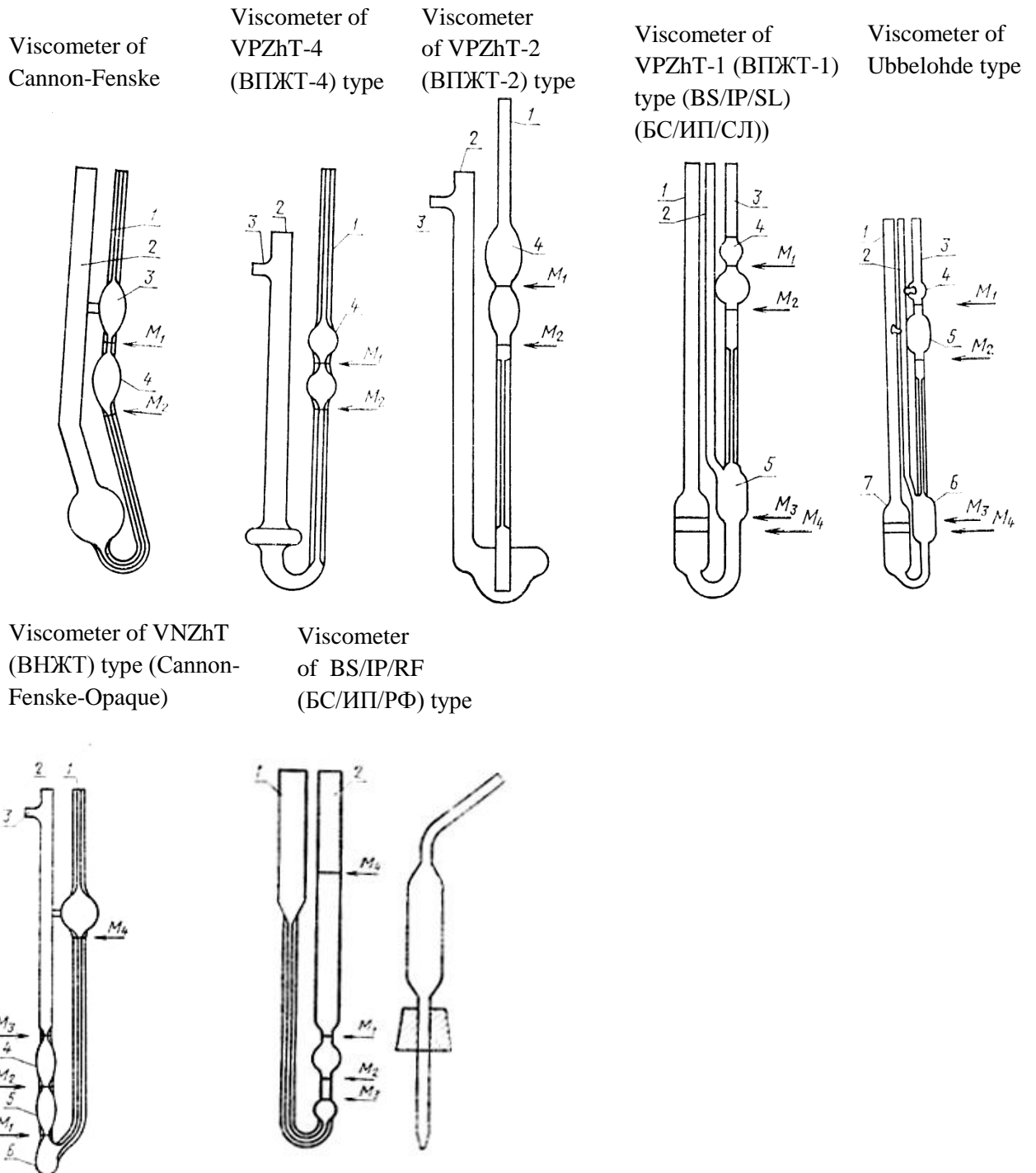


Figure 1 – Viscometers

3.3.3.2 The filled viscometer is kept in the thermostatic oven (bath) for 20–30 min to reach a temperature equilibrium. Where so required by the viscometer design, after the tested product reaches the test temperature, product volume is brought up to a required level. If one thermostatic oven (bath) is used for several viscometers, it is not allowed to dip or withdraw viscometers from the thermostatic oven (bath) while at least one viscometer is in the working state.

3.3.3.3 Using suction or pressure, bring the height of PMS (ПМС) fluid column in the viscometer capillary to approximately 5 mm above the first mark.

Measure the time of meniscus displacement from the first mark to the second, with the product effusing freely, to an accuracy of 0.2 s.

3.3.3.4 When using Ostwald viscometers and viscometers with hanging level, the effusion time shall be measured at least three times.

If the results of three consecutive measurements do not differ by more than 0.2 %, viscosity-to-density ratio is calculated as an arithmetic average (item 3.3.4.1).

When using reverse-flow viscometers, make at least two consecutive measurements; the difference between those shall not exceed 0.35 % of the arithmetic average value.

In any other case the measurements shall be repeated.

3.3.4. Processing of Test Results

Calculate viscosity-to-density ratio of the tested product ν , mm^2/s , using the following formula:

$$\nu = C \cdot \tau,$$

where C – viscometer constant, mm^2/s^2 ;

τ – arithmetic average time of polymethylsiloxane fluid effusion in viscometer, s.

The results of viscosity-to-density ratio determination are rounded to 0.01 % of the measured or calculated value (respectively).

The result of the test is represented by an arithmetic average of the results of viscosity-to-density ratio measurement in two viscometers if the difference between them does not exceed the values given in Table 3.

Table 3

Viscosity measurement temperature, °C	Permissible difference, %, of the arithmetic average value for viscometers of type		
	VPZh-1 (БПЖ-1)	VPZh-2 (БПЖ-2) VPZh-4 (БПЖ-4)	VNZh (БНЖ)
from -60 to -30	±2.0	±2.5	±3.0
from -30 to +15	±1.2	±1.5	±2.0
from +15 to +150	±1.0	±1.2	±1.5

3.4 Dynamic Viscosity Determination

The principle of the method consists in recording an antitorque moment of inner cylinder or cone of the measuring device with the tested product under different shear rates and calculation of the shearing stress and dynamic viscosity.

3.4.1 Equipment, Materials and Reagents Applied

A viscosity measuring device, comprising:

Rotary viscometer;

Resistance thermometer and measurement bridge of class not lower than 0.1 to control temperature in the cone – plate measuring device;

Liquid-circulating thermostatic oven providing temperature from minus 60 to plus 20 °C with an error not greater than 0.5 °C;

Cooling vessel;

Relay valve;

Contact thermometer for measuring temperature from minus 58 to plus 30 °C with division value of 1 °C;

Control thermometer for circulating thermostatic ovens to measure temperatures from minus 60 to plus 30 °C with division value of 0.5 °C;

A set of control thermometers for thermostating chambers of cylindrical measuring devices to measure temperatures from minus 60 to plus 30 °C with division value of 0.5 °C;

Measuring unit to record antitorque moment.

Dewar vessel or storage container of any type for dry ice.

Naphta for rubber industry applications.

Rectified industrial ethyl alcohol or industrial ethyl alcohol.

Solid carbon dioxide.

Conical flask of 250 cm³ capacity made of thermal-resistant glass.

Laboratory filter paper.

Funnel of dia. 100 mm, height 150 mm or 200 mm, made of chemically resistant glass.

Absorbent cotton wool.

Drying oven providing steady temperature with an error of 5 °C, maximum.

Beaker of 50 or 150 cm³ capacity.

Rubber squeeze bulb.

Stopwatch.

3.4.2 Test Preparation

3.4.2.1 Mix a polymethylsiloxane fluid sample thoroughly and filter through filter paper. For testing in a cylindrical device S₃, prepare 120 cm³ of tested product, and a cylindrical device H – 50 cm³ of tested product.

3.4.2.2 Pour the tested product into a beaker, heat in the drying oven up to 60–70 °C, and keep at this temperature for 15–20 min, while stirring from time to time.

3.4.2.3 Take the heated beaker with the tested sample out of the drying oven and cool down to the ambient temperature without stirring.

3.4.2.4 Prepare viscosity measuring device in accordance with its operation manual.

3.4.2.5 Wash all parts of the measuring devices with solvent, dry the parts and assemble the devices.

3.4.2.6 Fill an assembled measuring device with the tested polymethylsiloxane fluid sample at the ambient temperature in accordance with device operation manual.

3.4.2.7 Before carrying out further tests, thoroughly remove moisture from the working surfaces of viscometer after they cool down to the ambient temperature.

3.4.3 Test Procedure

3.4.3.1 Connect measuring device to the thermostatic oven, cool down to a temperature of minus 50 °C at the rate of 1–2 °C per minute, and keep at this temperature for 30 min with an error not greater than 0.5 °C.

3.4.3.2 Carry out the test in accordance with viscometer operation manual.

3.4.3.3 Record a relative rotation angle α of the inner cylinder or cone at a temperature of minus 50 °C.

3.4.3.4 A steady-state value that remains unchanged for 1 min shall be assumed as a test result. If the value of α has not settled, take the reading after 3 min.

3.4.3.5 Repeat the test with a new portion of the tested product as per items 3.4.2.4–3.4.3.4.

3.4.4 Processing of Test Results

3.4.4.1 An arithmetic average value of two consecutive measurements shall be assumed as a test result.

3.4.4.2 Dynamic viscosity η , Pa · s, is calculated using the following formula:

,

where τ – shearing stress, Pa;

D – shear rate, s⁻¹

3.4.4.3 Shearing stress τ , Pa, is calculated using the following formula:

$$\tau = \frac{Z \cdot \alpha}{10},$$

where Z – measuring device constant specified in the certificate, dyn/cm² · scale division;

α relative rotation angle on the measuring unit, scale divisions;

10 – conversion factor for conversion of the measuring device constant into SI units.

3.5 Determination of Flash Point in Open Cup

Determination of flash point in an open cup is done using Cleveland open-cup method (Method A) and Brenken method (Method B).

The principle of the methods consists in heating a sample of the tested product in an open cup at a specified rate until ignition (flash point) of product vapors occurs over product surface, caused by an incendiary device, and until

ignition (fire point) of the product occurs in the course of continued heating, with burning duration of at least 5 s.

3.5.1 Equipment, Materials and Reagents Applied

Apparatus for determining flash point and fire point in an open cup of type TVO (TV-2) (TBO (TB-2)) or semi-automatic and automatic types ATVO (ATV-2) (ATBO (ATB-2)), yielding results within the limits of tolerable differences as per the Cleveland method.

Three-leaf screen painted black on the inside, with sections of (46 ± 1) cm in width and (60 ± 5) cm in height, or a shield 55–65 cm high made from steel roofing sheet and painted black on the inside.

Thermometer.

Stopwatch of any type.

Mercury barometer or aneroid barometer with a measurement error not greater than 0.1 kPa.

Laboratory filter paper.

Pipette.

Wire brush.

Naphtha with boiling range from 50 to 170 °C, or nefras S50/170 (нефрас C50/170).

Drying agents (dehydrated): anhydrous sodium sulfate or industrial sodium sulfate, or industrial calcium chloride, or sodium chloride.

Distilled water.

Additionally for Method B.

Apparatus for determining flash point and fire point in an open cup as per the Brenken method (of type LTVO (JTBO)).

3.5.2 Method A

3.5.2.1 Test Preparation

3.5.2.1.1 Mix a sample thoroughly and carefully.

A test product containing water shall be dried at room temperature by shaking, with addition of one of the drying agents.

3.5.2.1.2 Install apparatus on a flat desk in such a place where there is no noticeable air movement and a flash can be clearly visible. To protect the apparatus from air movement, surround it from three sides with a screen or a shield. Cool the apparatus before each test.

3.5.2.1.3 Wash the cup with solvent before each test. Remove carbonaceous deposits with a wire brush. Then wash the cup with cold distilled water and dry it on an open flame or a hot plate. Cool the cup down to a temperature which is lower than the expected flash point by at least 56 °C and place it into the apparatus.

Place a thermometer into the cup in upright position, so that the lower end of the thermometer is at a distance of 6 mm from the cup bottom and equidistant from the cup center and walls.

3.5.2.2 Test Procedure

Fill the cup with polymethylsiloxane fluid, so that the upper meniscus exactly coincides with the mark. If the cup is filled above the mark, remove excessive polymethylsiloxane fluid with a pipette or other suitable appliance. Remove air bubbles from the sample surface. Wetting of the cup walls above fluid level is not allowed.

If tested product gets onto the outer walls of the cup, remove the product from the cup and treat the latter according to item 3.5.2.1.3.

Heat the cup with sample with the flame of a gas burner or by means of electric heating at a rate of 14 to 17 °C per minute in the first instance. When sample temperature becomes lower than the expected flash point by approximately 56 °C, adjust the heating rate, so that during the final 28 °C before the flash point the tested product is heated at a rate of 5 to 6 °C per minute.

Light up incendiary device flame and adjust it in such a way that flame diameter is about 4 mm. Compare flame size against a template (gauge ball) integrated in the apparatus.

Starting from a temperature which is lower than the flash point by at least 28 °C, use the incendiary device each time the sample temperature increases by 2 °C. Move the incendiary device flame in the horizontal direction, without stopping above cup edge and tracing the flame over the cup center in one direction for 1 s.

In the course of further temperature rising, move the igniting flame in the opposite direction.

Assumed as the flash point is the temperature indicated by the thermometer at the moment of the first blue flame appearing on part of the surface or on the entire surface of the tested product.

In case of an uncertain flash appearing, it shall be confirmed by the following flash after 2 °C.

A blue circle (halo) which is sometimes formed around the ignition flame is disregarded.

To determine the fire point, continue sample heating at a rate of 5 to 6 °C per minute and repeat testing with the incendiary device flame after each 2 °C of tested product temperature increase.

Assumed as the fire point is the temperature indicated by the thermometer at the moment the tested product ignites from the incendiary device flame set to it and keeps on burning for at least 5 s.

3.5.2.3 Processing of Test Results

If during the test the barometric pressure is below 95.3 kPa (715 mm Hg), introduce corresponding corrections as per Table 4 into the obtained flash point and fire point values.

Table 4

Barometric pressure, kPa (mm Hg)	Correction, °C
From 95.3 to 88.7 (from 715 to 665)	+2
From 88.6 to 81.3 (from 664 to 610)	+4
From 81.2 to 73.3 (from 609 to 550)	+6

An arithmetic average value of the results of two measurements rounded to an integer number and expressed in Celsius degrees shall be assumed as a test result.

3.5.3 Method B

3.5.3.1 Test Preparation

Test preparation shall be as per item 3.5.2.1.

3.5.3.2 Test Procedure

Cool the cup down to a temperature within 15 to plus 25 °C and place it into the apparatus's outer cup with annealed sand, so that the sand is at a height of 12 mm from the inner cup edge and there is sand between this cup bottom and the outer cup, with sand layer thickness being within 5 to 8 mm, which shall be checked with a template.

Place a thermometer in the upright position into the cup with tested product, so that the mercury bulb is in the cup center, approximately equidistant from the cup bottom and from the tested product level, and secure in this position in the support clamp.

Pour the tested product into the inner cup, so that fluid level is 18 mm away from the cup edge.

Check correct pouring of the tested product with a template; pour the tested product until tested product surface coming into contact with nib point of the fluid level height indicator.

During pouring, do not allow sputtering of the tested product and wetting of the cup walls above fluid level.

Heat the outer cup of the apparatus with the flame of a gas burner or Barthel lamp, or by electric heating, so that the tested product is heated by 10 °C per 1 min.

With 40 °C remaining to the expected flash point, reduce heating to 4 °C per 1 min.

With 10 °C remaining to the expected flash point, slowly trace the incendiary device flame along the cup edge at a distance of 10–14 mm from the tested product surface and parallel to it. Flame length shall be within 3–4 mm. The time of flame movement from one side of the cup to the other shall be 2–3 s.

Such test shall be repeated every 2 °C of temperature increase.

Assumed as the flash point is the temperature indicated by the thermometer at the moment of the first blue flame appearing on part of the surface or on the entire surface of the tested product.

In case of an uncertain flash appearing, it shall be confirmed by the following flash after 2 °C.

Reflection from the incendiary device flame should not be mistaken for a true flash.

To determine the fire point, continue heating of the outer cup in such a way that the product is heated at a rate of 4 °C per minute and repeat testing with the incendiary device flame after each 2 °C of tested product temperature increase.

Assumed as the fire point is the temperature indicated by the thermometer at the moment the tested product ignites from the incendiary device flame set to it and keeps on burning for at least 5 s.

3.5.3.3 Processing of Results

Processing of the results is done as per item 3.5.2.3.

3.5.4 Method Accuracy

3.5.4.1 Repeatability

Two results of flash point measurement obtained by one person in one laboratory shall be regarded as reliable (with 95 % confidence factor) if the difference between them does not exceed 4 °C.

The difference between two consecutive measurements of fire point shall not exceed 6 °C.

3.5.4.2 Repeatability

Two test results obtained in two different laboratories shall be regarded as reliable (with 95 % confidence factor) if the difference between them does not exceed 16 °C.

3.6 Setting Point Determination

The methods consist in preheating a test specimen of petroleum product and its subsequent cooling down at a specified rate to a temperature at which the sample remains immovable.

3.6.1 Method A

3.6.1.1 Equipment, Materials and Reagents Applied

Flat-bottomed test tube of transparent glass, inside diameter from 30.0 to 33.5 mm, height from 115 to 125 mm.

The outer surface of the test tube shall have three indelible marks:

Middle – corresponding to 45 cm³ sample volume, upper, and lower, indicating permissible deviations of sample volume, which make ± 3 cm³;

Flat-bottomed glass or metal ferrule, height from 113 to 115 mm, with inside diameter by 9.5–12.5 mm larger than outside diameter of the flat-bottomed test tube;

Cork or felt disk, 6 mm thick and with diameter corresponding to the inside diameter of ferrule;

Ring-shaped gasket, about 5 mm thick, tightly adjacent to test tube outer surface and freely entering the ferrule. The gasket can be made of natural cork or felt. Its purpose is to prevent test tube from touching ferrule;

Natural-cork or rubber plug corresponding to test tube inside diameter, with a hole for thermometer in the middle;

Mercury-filled thermometer with measurement limit from minus 30 to plus 60 °C;

Thermometer of any type for measuring coolant temperature, with calibration of scale corresponding to 1 °C;

Bath of arbitrary shape and size, with required bath temperature maintained with the help of condenser of cooling mixture;

Molten calcium chloride.

The cooling mixtures applied are as follows:

Water and ice – for temperatures above 0 °C;

Sodium chloride (NaCl) and ice – for temperatures up to minus 20 °C:

Denaturated alcohol;

Reclaimed ethyl alcohol and carbon-dioxide ice or other cooling mixtures – for temperatures below minus 20 °C.

3.6.1.2 Test Preparation

3.6.1.2.1 In the presence of water, the product shall be dehydrated. In case of considerable amount of water, remove it through presedimentation followed by product draining.

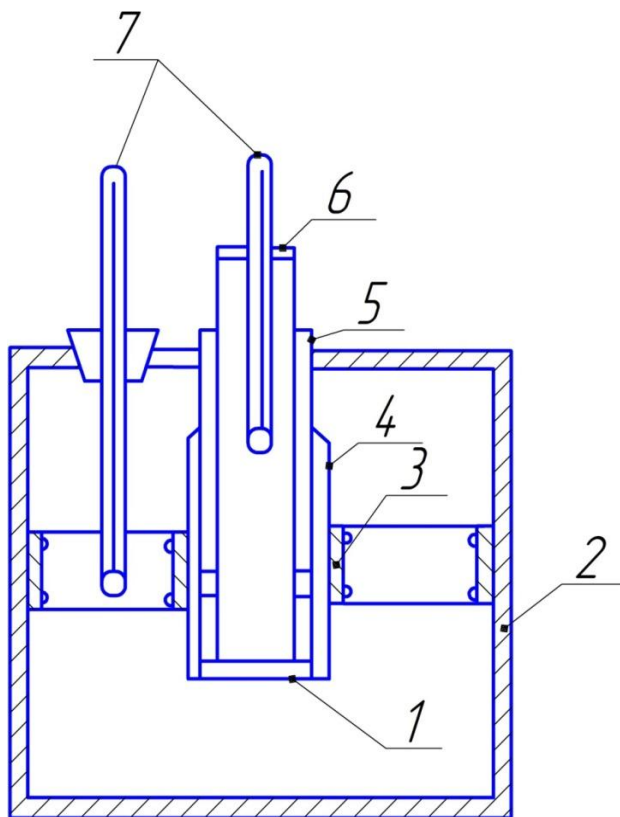
After that, agitate the product periodically for 10–15 min with freshly incinerated and ground sodium sulfate or calcium chloride, then settle the product and filtrate through a dry filter.

3.6.1.2.2 Pour the dehydrated product into a dry clean test tube up to one of the volume marks, so that the product does not spread over test tube walls. If necessary, heat up the product (but not above 45 °C) on a water bath to the liquid state and transfer it to test tube.

3.6.1.2.3 If it is known that over the preceding 24 hours the sample was subjected to heating above 45 °C or if its previous storage temperature conditions are not known, the sample shall be kept at the ambient temperature for 24 hours.

3.6.1.2.4 Tightly close the test tube with polymethylsiloxane fluid using a plug with an appropriate thermometer inserted into it for setting point determination.

The plug shall be tightly fitted to the test tube, so that thermometer axis coincides with that of the test tube. The upper part of thermometer bulb shall be immersed to 3 mm below polymethylsiloxane fluid surface (Figure 2).



1 – disk; 2 – bath; 3 – backing; 4 – test tube; 5 – ferrule; 6 – cork plug;
7 – thermometer

Figure 2 – Instrument for determination of setting point

3.6.1.2.5 The tested polymethylsiloxane fluid filled into test tube shall be subjected to pre-treatment, proceeding as follows: heat it on a water bath without stirring up to 45 °C.

3.6.1.2.6 Place disk on the ferrule bottom and put on a sealing ring on the flat-bottomed test tube at a height of 25 mm from the bottom.

The disk, the sealing ring, and the walls of ferrule and test tube shall be clean and dry. Insert test tube into ferrule.

3.6.1.2.7 Test Procedure

3.6.1.2.7.1 Prepare a bath with temperature within minus 1 to plus 2 °C and maintain this temperature throughout the test.

Install test tube with ferrule in the bath in the upright position, so that ferrule top is 25 mm above cooling mixture level.

During the test, do not allow thermometer shifting, so as to avoid violation of paraffine crystals structure. It is not allowed to place test tube into the cooling medium without ferrule.

3.6.1.2.7.2 The process of test tube taking out of and placing back in the ferrule shall take 3 s maximum.

If the tested specimen has not lost fluidity during cooling down to 9 °C, transfer the test tube to the ferrule of another bath, whose temperature shall be maintained within minus 18 to minus 15 °C.

If the product retains fluidity when its temperature reaches minus 6 °C, transfer the test tube to the ferrule of a bath where the temperature is maintained within minus (33 ± 1) °C.

When determining very low setting points, it is necessary to have the temperature of each next bath by 17 °C lower than that of the previous one. In each case, transfer the test tube with the product being tested to the next bath after the machinery oil specimen being tested has cooled down to a temperature which is by 27 °C higher than the temperature of the next bath.

Once it is noticed that, with the test tube tilted, the tested product remains immovable, set the test tube to horizontal position and observe product surface.

If within 5 s (determined with a stopwatch or other precision instrument) any shifting of the surface is noticed, immediately place the test tube with the tested product into ferrule and repeat fluidity test after additional cooling-down by 3 °C.

3.6.1.2.8 Processing of Test Results

The temperature at which the product remains immovable for 5 s, with the test tube set to horizontal position, shall be assumed as a setting point of the tested polymethylsiloxane fluid.

A permissible difference between parallel measurements shall not exceed 3 °C.

3.6.2 Method B

3.6.2.1 Equipment, Materials and Reagents Applied

Glass test tube with spherical bottom, height (160 ± 10) mm, inside diameter (20 ± 1) mm, with an indelible circumferential mark made on the outer side surface of the test tube at a distance of 30 mm from its bottom;

Test tube–ferrule with a concave or spherical bottom; test tube height (130 ± 10) mm, inside diameter (40 ± 2) mm;

Natural-cork or rubber plug corresponding to test tube inside diameter, with a hole for thermometer in the middle;

Mercury-filled thermometer with measurement limit from minus 30 to plus 60 °C;

Thermometer of any type for measuring coolant temperature, with calibration of scale corresponding to 1 °C;

Bath of arbitrary shape and size, with required bath temperature maintained with the help of condenser of cooling mixture;

Rectified ethyl alcohol with carbon dioxide ice. It is allowed to apply other agents providing specified cooling temperature.

3.6.2.1.2 Test Preparation

3.6.2.1.2.1 If there is water in polymethylsiloxane fluid, the latter shall be dehydrated before the test as per item 3.6.1.2.

3.6.2.1.3 Prior to the test pour 0.5–1.0 cm³ of sulphuric acid or fuming sulphuric acid into test tube–ferrule.

3.6.2.1.4 Pour the dehydrated product into a dry clean glass test tube up to the mark, so that the product does not spread over test tube walls. Using a cork plug, tightly insert into the test tube an appropriate thermometer, securing it in such a way that it is on the test tube axis and its bulb is at a distance of 8–10 mm from the test tube bottom. For better thermometer steadiness in the test tube when in working position, put a cork plug on thermometer lower part (approximately at its mid-length), fitted in such a way that it enters the test tube with a little friction.

3.6.2.1.5 Put the test tube with product and thermometer in a water bath preheated to a temperature of (50 ± 1) °C and hold it there until the product has the temperature of the bath.

3.6.2.2 Test Procedure

3.6.2.2.1 Take the test tube with product and thermometer out of the water bath, wipe dry on the outside, and secure it with the help of a cork plug in the test tube-ferrule in such a way that walls of the latter are approximately equidistant from ferrule walls. Secure the assembled device in support holder in the upright position and leave at room temperature for the polymethylsiloxane fluid to cool down to (35 ± 5) °C, then place fluid into a vessel with cooling mixture whose temperature shall be preset to a value which is by 5 °C below the setting point to be determined.

During product cooling down, the preset cooling mixture temperature shall be maintained within an error of ± 1 °C.

When product in the test tube has the temperature of expected setting point determination, tilt the device to an angle of 45° and, without taking it out of the cooling mixture, hold in this position for one minute.

After that, carefully take the device out of the cooling mixture, quickly wipe the test tube-ferrule and check if the tested product meniscus has shifted.

3.6.2.2.2 If the meniscus has shifted, take test tube out of the ferrule, heat it again up to (50 ± 1) °C, and carry out a new setting point determination at a temperature which is by 4 °C lower than the previous one, until the meniscus stops shifting at a certain temperature.

Before a new determination, in order to protect the device from damage caused by strong thermal effects, leave the test tube with product and thermometer at room temperature until the product has a temperature of 20 °C, and only thereafter place the test tube in a water bath.

3.6.2.2.3 If the meniscus has not shifted, take test tube out of the ferrule, heat it again up to (50 ± 1) °C, and carry out a new setting point determination at a temperature which is by 4 °C higher than the previous one, until the meniscus stops shifting at a certain temperature.

3.6.2.2.4 After finding the setting limit (transition from fluidity to immovability or vice versa), repeat determination, increasing or decreasing test temperature by 2 °C until such a temperature is established at which product meniscus remains immovable, whereas during a repeated test at a temperature by 2 °C higher, it shifts. This temperature shall be recorded as the one determined for a given test.

3.6.2.2.5 To determine product setting point, two parallel tests shall be carried out, with the second test started from a temperature which is by 2 °C higher than the temperature determined during the first test.

3.6.2.3 Processing of Test Results

An arithmetic average value of two parallel measurements shall be assumed as a setting point of the tested polymethylsiloxane fluid. The difference between the results of parallel measurements shall not exceed 2 °C.

3.7 Determination of Reaction of Medium (pH of Aqueous Extract)

3.7.1 To determine reaction of the medium, the following means are used:

Industrial ethyl alcohol with pH no lower than 6.7; it is prepared through distillation over sodium hydroxide or potassium hydroxide;

Hydrolytic ethyl alcohol for electrode washing;

Sodium hydrate (sodium hydroxide) or potassium hydrate (potassium hydroxide);

Potassium chloride, saturated solution;

Benzene carbonic acid for colorimetric determination, 0.1N solution in the solvent mixture of toluene and ethyl alcohol in the ratio of 7:3;

Solvent with pH (6.7 ± 0.1), prepared as follows: benzene carbonic acid solution is added by drops to a 7:3 mixture of toluene with absolute ethyl alcohol to obtain a required pH;

Hydrochloric acid, 0.1N solution;

Regular standard heptane, ch. p., or naphta for rubber industry applications, or toluene with pH 6.5–7.0;

Distilled water, double-distilled, pH 6.0–7.0;

Fixanal for preparation of standard buffer solutions for pH-metering;

Measuring cylinders of 25 and 100 cm³ volume;

Cylindrical separating glass funnels of 100 and 250 cm³ capacity, and conical funnels;

Flat-bottomed or conical flasks of 250 cm³ capacity;

Glass condenser, laboratory-type, with 6–10 balls;

Tall cups for weighing (weighing bottles);

pH-meter with measuring sensitivity of ± 0.01 pH, with glass and silver-chloride glass electrodes;

Magnetic agitator;

Hot plate of closed type.

3.7.2 Test Preparation

3.7.2.1 Preparation of pH-Meter and Electrodes

Prepare pH-meter and electrodes for operation in accordance with the attached instructions.

Then adjust the instrument as per standard buffer solutions with pH 4.01 and 6.86.

Check and adjust pH-meter daily before carrying out the tests.

3.7.3 Test Procedure

Enter 30 cm³ of tested product, 30 cm³ of heptane and 30 cm³ of double-distilled water into separating funnel and stir for 10 min, then, after 10–15-minute settling, drain aqueous extract into a beaker and determine pH in accordance with the instructions attached to the instrument.

3.7.4 Processing of Test Results

An arithmetic average value of two parallel measurements with the maximum permissible difference between them not exceeding 0.15 pH shall be assumed as a test result.

3.8 Determination of Silicium Weight Ratio

3.8.1 The principle of the method consists in product weight quantity decomposition in a quartz flask or porcelain crucible with 25-percent fuming sulphuric acid and fuming nitric acid containing 20 % of nitric oxides and incineration of the obtained silicium dioxide at 800 °C.

3.8.2 Equipment, Materials and Reagents Applied

25-percent fuming sulphuric acid, prepared by diluting 60-percent fuming sulphuric acid with sulphuric acid;

Sulphuric acid, analitically pure;

Nitrogen acid with 20 % of nitric oxides;

Conical quartz flask with ground plug, capacity 75–100 cm³, height 80–90 mm;

Straight drain-out measuring pipette, accuracy class 1, capacity 2–5 cm³, minimum division value 0.01 cm³;

Tall porcelain crucible, largest diameter 45 mm. It is allowed to use crucibles and cups made of platinum;

Exiccator with metal mesh;

Hot plate of closed type;

Muffle furnace providing temperature of 850 °C;

General-purpose laboratory balance, accuracy class 4, with the maximum weighing limit of 200 g.

It is allowed to use other measuring instruments and other equipment whose metrological characteristics and specifications, respectively, are no worse than those specified in this document, as well as other reagents with quality no lower than specified herein.

3.8.3 Test Procedure

Take for analysis 0.5 g of the analyzed polymethylsiloxane fluid, weigh it with an error of max. 0.0002 g in a quartz flask or porcelain crucible pre-incinerated in a muffle furnace at 800 °C until achieving a constant weight (permissible difference between two latest weighing cycles shall not exceed 0.0002 g), add into it 1.5 cm³ of 25-percent fuming sulphuric acid and 2 or 3 drops

of nitrogen acid, and carefully heat on a hot plate. Then again add nitrogen acid by drops into the flask until discoloration of the brown vapors stops, which points to the ending process of organic silicon compounds oxidation. After that, add 2 or 3 more drops of nitrogen acid and evaporate the content of flask or crucible, while increasing heating, to remove excess acid. During heating, watch out to avoid intense foaming. When foaming is about to start, remove flask or crucible from the hot plate.

Once vapor emission has stopped, place flask or crucible into muffle furnace and incinerate at a temperature within plus 800 to plus 850 °C for 2 hours. Then transfer flask or crucible to exiccator, cool down for 30–60 min, and weigh with an error not greater than 0.0002 g. Repeat incineration, cooling-down and weighing until a constant weight is obtained.

In the absence of nitrogen acid, take 4 cm³ of fuming sulphuric acid and hold for 1 hour, then proceed as described above.

Carry out a check test under the same conditions and with the same amount of reagents.

3.8.4 Processing of Results

Calculate the weight ratio of silicium X, %, using the following formula:

where m – tested product weight quantity, g;

m_1 – weight of remainder after incineration, g;

m_2 – weight of remainder in check sample after incineration, g;

0.4674 – scale factor in conversion from SiO₂ to Si.

An arithmetic average value of three parallel measurements with the maximum permissible difference between them not exceeding 0.5 % shall be assumed as a test result.

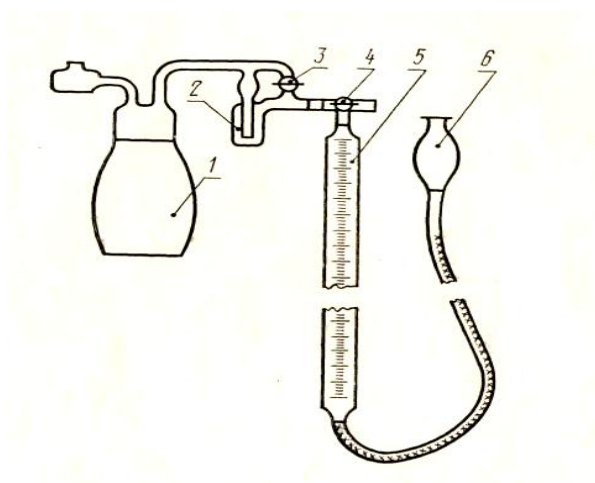
3.9 Determination of Water Weight Ratio

3.9.1 Method Principle

The principle of the method consists in interaction of potassium hydride with water contained in the fluid, followed by measurement of hydrogen released in the process.

3.9.2 Equipment, Materials and Reagents Applied

Device for determination of water weight ratio (Figure 3)



1 – reaction flask with an offshoot for calcium hydride; 2 – Drechsel flask;
3 – one-way cock; 4 – three-way cock; 5 – drop-meter; 6 – levelling bottle

Figure 3 – Device for determination of water weight ratio

Mercury-filled glass thermometer with measurement range from 0 to plus 50 °C, division value 0.5 °C.

Technical barometer.

General-purpose laboratory balance, accuracy class 4, with the maximum weighing limit of 500 g.

Sulphuric acid, analitically pure.

Calcium hydride.

Distilled water.

Vacuum grease.

3.9.3 Analysis Preparation

Fill gas drop-meter 5 and levelling bottle 6 (Figure 3) with distilled water, and fill Drechsel flask 2 (Figure 4) with sulphuric acid in such amount that the hollow ball end is dipped into the acid to 4–5 mm.

Lubricate the cocks with vacuum grease. Sulphuric acid in the Drechsel flask shall be changed after 20 measurements or after 15 days, whichever comes first. Check the assembled device for tightness proceeding as follows: set three-way cock of the drop-meter to a position in which drop-meter communicates with reaction flask (Figure 5) and atmosphere. By moving the levelling bottle, set water level in the drop-meter to zero.

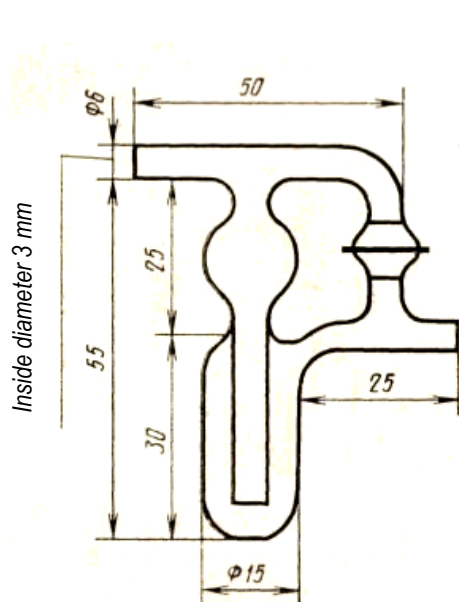


Figure 4 – Drechsel flask
with cock

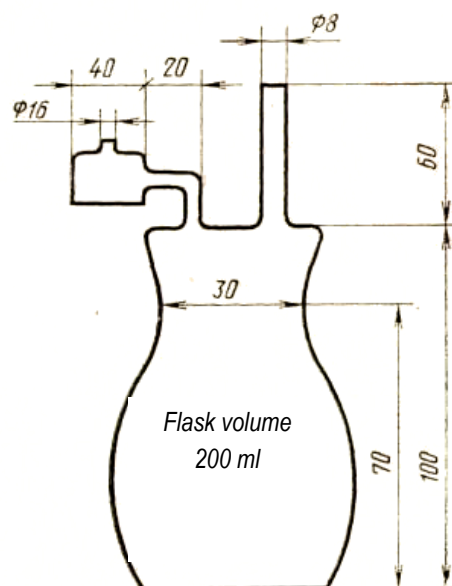


Figure 5 – Reaction flask

By turning three-way cock through 90 °C, isolate the device from atmosphere, lower the levelling bottle down to the level of drop-meter bottom and hold so for 5 min. Then, by raising the levelling bottle, equalize water meniscus in the drop-meter and the bottle; if in so doing water level in the drop-meter sets to zero, the device shall be considered leak-tight.

3.9.4 Analysis Procedure

The room where analysis is carried out shall be thermostabilized. Temperature variations over the time of one analysis shall not exceed 0.5 °C.

In the course of analysis, take barometric pressure and temperature readings.

Weigh reaction flask with plug with a maximum error of 0.1 g.

Place 100 g of the fluid being analyzed into reaction flask and weigh with the maximum error of 0.1 g, recording the weighing result in grams with the accuracy to the fourth decimal point.

Place about 1 g of powdered calcium hydride into flask offshoot. Then connect reaction flask to the device as shown in Figure 3 and set solution level in the drop-meter to zero, proceeding as described above.

After that, turn reaction flask in such a way that calcium hydride gets from flask offshoot into the fluid, and thoroughly mix flask content.

Collect released hydrogen into the drop-meter, while gradually lowering the levelling bottle. Shake the flask every 10–15 min. Observe water level in the drop-meter as water menisci in the levelling bottle and the drop-meter coincide. Take readings after 5 min from flask shaking.

Determination is considered finished when two readings taken 15 min apart coincide. Analysis duration from the moment of calcium hydride pouring into the fluid until the last reading shall be no less than 60 min.

3.9.5 Processing of Results

Water weight ratio X_1 , %, shall be calculated using the following formula:

where V – volume of hydrogen released in the course of analysis, cm^3 ;

P – barometric pressure, Pa (mm Hg);

p – water vapors pressure at analysis temperature, Pa (mm Hg);

0.000804 – scale factor for hydrogen conversion into cm^3 , normalized to temperature $0\text{ }^\circ\text{C}$ and barometric pressure 101 325 Pa (760 mm Hg) per water weight, g;

t – analysis temperature, $^\circ\text{C}$;

m – weight quantity of the fluid analyzed, g.

An arithmetic average value of two parallel measurements, with the permissible difference between them not exceeding 0.001 %, shall be assumed as an analysis result.

4 Packing, Marking, Transportation and Storage

4.1 Polymethylsiloxane fluid is packaged into tin-plate cans of 10 dm^3 capacity, metal canisters for petroleum products, glass bottles, zinc plated flasks, drums of zinc plated steel with longitudinal welded and transverse rolled seams. Upon agreement with the customer, it is allowed to package fluids into containers made of the same materials with capacity less than 10 dm^3 .

The neck of canister or can shall be soldered or sealed off using another method providing complete tightness of the canister or can.

The necks of bottles shall be wrapped with moistureproof film and bound with cord.

Container with the fluid shall be sealed.

4.2 Each production unit shall be provided with a label, or stenciled designations shall be made in waterproof paint, indicating the following:

- Manufacturer's name and trademark;
- Product name;
- Manufacture date;
- Batch number;
- Gross weight and net weight;
- Designation of the present standard.

Shipping marking shall be made with application of handling signs: “This side up”, for glass bottles – “Fragile. Handle with care”. Polymethylsiloxane fluid is not a hazardous cargo.

4.3 Polymethylsiloxane fluid can be transported in closed transport vehicles of any type in accordance with the shipping rules applicable for a given means of transport.

Shipment can be done in the palletized form or in containers. Arrangement of freight pieces into unitized load blocks is done on flat pallets of 1 t bearing capacity, using metal band or wire for bracing load blocks and applying cardboard pads or metal belts.

Load block weight – 400 kg. The pattern of freight pieces arrangement in load blocks is in two tiers of 6–8 canisters each.

4.4 Polymethylsiloxane fluid shall be stored in the manufacturer's packaging in an indoor storage facility.

5 Manufacturer's Warranty

5.1 The manufacturer guarantees fluid compliance with the present standard provided that transportation and storage conditions are observed.

5.2 Shelf life of the fluid is 5 years from the manufacture date.

6 Safety Requirements

6.1 Polymethylsiloxane fluid PMS-200 (ИМС-200) is chemically inert. In terms of effect on the human system, it is a low-hazard substance (class IV). It exerts no toxic effect on skin and conjunctiva.

6.2 Polymethylsiloxane fluid is an explosion-proof combustible substance. Depending on its viscosity, it has the following inflammation temperature limits of vapors in the air:

lower – from plus 128 to plus 214 °C,

upper – from plus 256 to plus 297 °C.

Spontaneous ignition temperature of the fluid is within plus 330 to plus 400 °C, depending on its viscosity.

6.3 Given that the fluid is flammable, use of open flame when handling it is prohibited. When burning, the fluid decomposes with release of carbon dioxide and silicium dioxide.

6.4 Fire fighting means for the fluid: sand, felted cloth, carbon-dioxide fire extinguisher OU-2 (OY-2).

6.5 The fluid shall be handles in an exhaust hood, with exhaust ventilation provided. The individual protective equipment to be used shall include rubber gloves and protective goggles.