RUSSIAN FEDERATION

JSC “VOLZHSKY ORGSYNTHESE”

STANDARDS OF ORGANIZATION

STO 00204168-001-2008

N-METHYLANILINE TECHNICAL IMPROVED

Specifications

2008

Volzhsky, Volgograd region

Information about the standard

1 DEVELOPPED BY

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2 AGREED BY

JSC “All-Russian Research Refining Institute” (letter №23/44-2798 of 06.09.2007).
“Komsomolsk Refinery” Ltd. (letter №14/7337 of 04.10.2007).
“Serpoukhovsk Fuel Company” Ltd. (letter № 229 of 05.10.2007).
JSC “Achinsk Refinery of the Eastern Fuel Company” (letter №202/4228 of 08.10.2007).
JSC “Khabarovsk Refinery” NK “Alliance” (letter № 34 of 11.10.2007).
JSC “Angarsk Petrochemical Company” (letter № 227-к-20374 of 16.10.2007).
“Expert Car-building Center” Ltd. (expert conclusion № 53601-08/ЭЦ of 23.04.2008).
Department of Infrastructure and Transportation of the Federal Agency of rail transport (letter № УИП-3/818-ш of 03.07.2008)
Department of commercial operation for goods transportation of JSC “Russian Railways” (letter № ЦМЭ-5/ 85 of 22.07.2008)

3 APPROVED AND IMPLEMENTED

By the order of General director of JSC “Volzhsky Orgsynthese” № 518 of July 16,
Implemented from **25.07.2008**

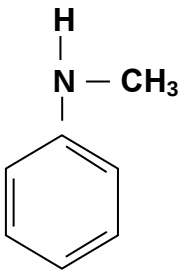
4 The standard consists of the following:

- chapter 3 of the Federal law of December 27, 2002 № 184 - ФЗ «About technical regulation»;
- clauses of GOST R 1.4-2004. Standardization in the Russian Federation. Standards of organization. General clauses (except points 4.15 and 4.12, which are realized incompletely);
- clauses of series GOST R ISO 5725 «Accuracy (correctness and precision) of measuring methods and results»;
- clauses of the Agreement on International Goods Transport by Rai (SMGS);
- clauses of the European Agreement concerning the International Carriage of Dangerous Goods by Road (DOPOG)

5 IMPLEMENTED

Instead of TU 2471-269-00204168-96 «N-methylaniline technical. Technical conditions».

6 REVISION with Amendment No. 1 of 24.10.2012



C_7H_9N / $C_6H_5NHCH_3$

N – methylaniline technical improved

Synonyms: N -monomethylaniline. N - methylphenylamine. N – phenylmethylamine. N – methylbenzolamine. (Methylamino)benzene.

IUPAC name # N-methylaniline
CAS # 100-61-8
RTECS # BY4550000
EINECS # 202-870-9
UN /OOH # 2294
ICSC CARD # 0921

Relative molecular weight # 107,15

Application

Antiknock additive to motor gasoline. It's applied for motor gasoline production to obtain appropriate detonation characteristic of gasoline and to correct octane number including production of ecologically clean gasoline.

It's the main compound of multifunctional octane-raising additives to motor gasoline.

1 Specification

1.1 Production form: oily transparent liquid with persistent smell of aromatic amino-compounds.
1.2 Physicochemical properties are conformed to standards indicated in table 1.

Table 1

Indices	Standard	Measurement method
1 N-methylaniline content, %	≥ 98,0	Supplement А
2 Aniline content, %	≤ 0,3	Supplement Б
3 N,N-dimethylaniline content, %	≤ 1,6	
4 Water content, %	≤ 0,1	Point 2 GOST 14870 Supplement В
5 Color (D)	≤ 0,100	Supplement Г

1.3 Octane number of mixture of isooctane and normal heptane taking at the ration (70:30) by volume is increased not less than by 6 units at the addition of 1,5% mass of N-methylaniline.
1.4 Additives with anti-oxidation, detergent and anti-corrosion properties may be added to the product upon the customer request. Name and content of additives are indicated in quality certificate.
1.5 Shelf life is 6 months since the production date if storage conditions are kept.

2 Packing, marking

2.1 *Packing*
2.1.1 Containers КСГМГ 1000МСК(1000МК) for transportation of hazardous liquids according to TU 2297-020-17152852-01.
Container consists of a steel welded framework and a rigid plastic internal tanks with service equipment, capacity (950±20) dm^3. Code container identification is 31HA1 according to the international classification.
Filling container level – not more than 94%.
2.2 *Marking*
2.2.1 Marking is printed on a label according to GOST 14192. Variable data are indicated during the batch production.
Other method of marking is possible.

2.2.2 Marking consists of the following obligatory information:
- manufacturer identification (name and logotype of the company, legal address, country, phone and fax numbers);
- product identification (product name, document name);
- product information (application, main properties, shelf life);
- batch information (number, production date, weight net);

- danger symbol  («Poison») according to GOST 19433 (drawing 6a) and classification code 6113, hazardous cargo number according UN classification # 2294;

- manipulation symbol  («Hermetic packing») according to GOST 14192 (fig. B.7).

Additional information is indicated upon the customer request.

3 Safety requirements

3.1 N-methylaniline is related to highly hazardous substances upon the exposure level on an organism, hazard class – 2 according to GOST 12.1.007.

It's a methemoglobin former. It causes poisoning at inhalation of high vapors concentrations, ingestion and skin absorption.

Product penetrates via intact skin causing general toxic effect. It's an eyes irritant. Repeated contact may cause the allergenic reaction on skin.

Extreme admissible concentration (EAC) of working zone is 0,2 mg/m^3 [1].

Extreme admissible concentration of ambient air in populated areas is 0,04 mg/m^3 (hazard class 3, limited hazard index – reflex effect) [2].

Extreme admissible concentration of water in household water and community use is 0,3 mg/l (hazard class 2, limited hazard index – organic odor) [3].

3.2 N-methylaniline is an inflammable liquid. Steams form explosive mixtures with air at temperature more than 80 ^{0}C.

Suitable extinguishing means: air mechanical foam, powder, carbon dioxide, water of aerosol dispersion. Extinguishing by compact water jets, water with moistening, water-alkaline solutions is not recommended.

3.3 When work with N-methylaniline, it's necessary to use protective clothes and shoes (cotton suit, rubber boots or lather shoes), hermetic goggles, rubber gloves or rubberized gauntlets. In emergency situation and in case of big spillages it's necessary to have a respirator with filter ДОТ^{TM}.

(Revised, Amendment № 1).

3.4 In case of non-keeping of handling, storage and transportation requirements, non-organized wastes placements, burning or disposal and in emergency situations, N-methylaniline contaminates ambient air, causes adverse effect on water reservoirs.

3.5 Detailed description of hazard effects on human and environment, their occurrence conditions, first aid measures, accidents prevention measures, individual protection means and other information is available in Safety Data Sheet, emergency cards and international information safety card (ICSC # 0921).

4 Transportation

4.1 N-methylaniline is transported by all transport means according to the rules of cargo transportation valid for concrete transport mean.

4.2 N-methylaniline is transported by railway:

- in special railway tanks-cars of the consigner (consignee) or in leased tanks-cars from carbon steel or aluminium, hermetically closed with top discharge according to "The Rules for Transportation of Liquid Goods in Bulk in tanks-cars and in Hopper wagons for transportation of oil bitumen" [4];

- in approved special containers-cisterns of the consigner consignee) or in leased containers-cisterns from carbon steel or aluminium without bottom discharge according to GOST 31314.3 (ISO 1496.3) or СК-5Ц according to GOST 30302 according to "The Regulations concerning the International Carriage of Dangerous Goods by Rail" [5] and "Technical conditions of cargo placement and fastening in wagons and containers" [6];

- in approved portable cisterns of type T4 of the consigner (consignee) or in leased cisterns from carbon steel or aluminium according to "the Regulations concerning the International Carriage of Dangerous Goods by Rail" [5] and "Technical conditions of cargo placement and fastening in wagons and containers" [6].

N-methylaniline transportation is possible in approved containers-cisterns and portable cisterns of other types having higher minimum test pressure and thicker boiler wall as well as stricter requirements as per holes located below liquid level and devices of pressure release.

Product filling (discharge) is carried out in specially conditioned places of the consigner (consignee) on tracks for non-general use.

Filling level of reservoirs – 94%, not more.

Product transport name – N-METHYLANILINE, dangerous cargo number according to UN classification # 2294, danger class 6 (subclass 6.1, classification code 6113) according to classification GOST 19433 and Standard rules of UN Recommendations concerning the International Carriage of Dangerous Goods.

4.3 Internationally N-methylaniline is transported by railway according to the Regulations concerning the International Carriage of Dangerous Goods to SMGS [7] (code of Harmonized Nomenclature Cargoes GNG-

292142, classification code T1, subclass 6.1, dangerous cargo code 60 (poisonous substance), packing group III (substance with low danger level), emergency card # 608.

N-methylaniline is transported in special railway tanks-cars or portable cisterns of the consigner (consignee) or in leased tanks-cars meeting the Supplement 2 to SMGS.

4.4 N-methylaniline transportation by trucks is carried out:

- in built cisterns (tanks trucks of type petrol tankers, oil tankers)
- in containers КСГМГ 1000МСК(1000МК) for hazardous liquids transportation by trucks.

N-methylaniline transportation by trucks is carried out according to “The Regulations concerning the International Carriage of Dangerous Goods by Road” [8] and DOPOG [9].

Dangerous cargo code 60, transport category of dangerous cargo according {9} – 2.

Transportation through tunnels of category E is prohibited.

4.5 Cisterns marking is effected according to the rules prescribed for every concrete transport mean (transport tare).

Marking of railway tanks-cars and containers-cisterns is carried out according to the requirements of [4] and [5], marking of tanks trucks – according to [9] indicating UN number 2294, danger code 60 and danger symbols “Poison” according to GOST 19433 (drawing 6a) and “Hermetic packing” according to GOST 14192 (fig. B.7).

4.4, 4.5 (Revised, Amendment № 1).

5 Storage

5.1 N-methylaniline storage is performed by the product manufacturer/consignee.

It’s recommended to store N-methylaniline in sealed steel reservoirs under nitrogen in open areas or in covered cool places equipped by combined extract and input ventilation. Product temperature during its storage has not to exceed 40 ^{0}C.

During the product storage without nitrogen, the color may be changed from yellowish up to light-brown under the influence of atmospheric oxygen.

For outdoor storage it’s recommended to use reservoirs like as stationary steel vessels for petroleum and its products.

Filling level of reservoirs – 94%, not more.

5.2 Storage with strong oxidants, strong acids, alkalis, foodstuffs, other consumption goods, animal foodstuffs is prohibited.

5.3 Guarantee shelf life is 6 months since the production date.

6 Product acceptance

6.1 N-methylaniline is accepted by batches.

Any quantity of homogenous product, drawn by one quality document (certificate) is considered as a batch. Quality certificate is stamped by Quality Control Department.

During the product shipment in cisterns (containers-cisterns), content of each transport mean is considered as a batch.

(Revised, Amendment № 1).

6.2 N-methylaniline is subject to acceptance and qualification tests.

Each product batch is subject to acceptance tests. Acceptance test includes the determination of standards indicated in table 1.

Qualification tests are carried out at the product implementation, replacement of normative documents for product and change of its production technology. Octane number increase with N-methylaniline addition is determined during qualification tests.

6.3 Samples are taken from each transport mean to check product quality conformance to this standard requirements.

If transported in composite containers, samples are taken from three ones, not less, provided that the batch consists of 20 packing units, not less.

6.4 In case of negative test results even for one standard, it’s necessary to retest the same sample in double volume from the same batch.

Retest results are final and applied to the whole batch.

6.5 Sampling from transport means is carried out according to GOST 6732.2 by a metal sampler or using close metal cans (cylinders) with covers or caps having an element to take sample at any vessel layers, from composite containers – by a glass dropping tube. Cans (cylinders) must have a handle length permitting to take a sample from the lowest vessel layer.

Point samples are taking from three layers. Average sample volume – not less than 250 cm$^{3.}$

Sampling according to GOST 2517 is possible at product acceptance by the customer.

6.3-6.5 (Revised, Amendment № 1).

6.6 N-methylaniline property to increase octane number not less than by 6 units is confirmed by the protocol of qualification test and ensured by the manufacturing process.

6.7 Assay content of each additive indicated in quality certificate is ensured by the manufacturing process and confirmed by results of operational control.

7 Tests methods

7.1 Acceptance tests are performed based on methods stipulated in Table 1 of the present standard.

It's possible to use alternative methods, measuring devices and equipment allowing to obtain metrological characteristics indicated in the standard as well as to apply other materials and reagents analogue by properties to stated ones.

If a chromatograph is equipped by a PC, chromatograms processing is carried out using an established program.

Methods of the present standard are referee, except determination of water content..

Method stipulated in point 2 of GOST 14870 is referee to determine water content. Mass of weighted amount used for this method is 1,0000 – 5,0000 g.

7.2 Octane number increase of mixture of isooctane and normal heptane taking at the ration (70:30) by volume at the addition of 1,5% mass of N-methylaniline is determined according to GOST 511.

7.3 Determination of additive (additives) content is fulfilled according to the internal method of the enterprise.

7.4 Quality control of measurement results is effected according to recommendations MI 2335 or RMG 76.

7.5 Conditions for measurements fulfillment:

- temperature……..	from +15 ˚C till +25 ˚C;
- relative air humidity…….	80 %, not more;
- atmospheric pressure……	84,0 - 106,7 KPa (630-800 Mm hg.)
Supply voltage…….	220^{+20}_{-33} V
Alternating current frequency……...	50±1 Hz

7.6 Measurements are carried out by the lab analyst of 4^{th} class, not lower.
(Revised, Amendment № 1).

Supplement A
(mandatory)

Determination of N-methylaniline content

A.1 Assigned characteristics of bias and measurements range

Assigned characteristics of bias method for probability level P = 0,95 and measurements range are given in the table A.1.

Table A.1

Index	Measurements range, %	Relative value	
		Accuracy factor $\pm \delta$, %	Reproducibility limit $R_{отн.}$, %, m = 2
N-methylaniline content	94,0 - 99,5 inclusive	0,3	0,4

A.2 Summary of test method

N-methylaniline content is determined by an indirect method based on deduction of organic impurities and water, defined by gas chromatography, from 100% contents sum.

A.3 Measurements

Aniline, N,N-dimethylaniline and non-identified impurities contents are measured by gas chromatography using a flame-ionization detector and water content is measured by gas chromatography using a thermal conductivity detector or by Fisher's method according to GOST 14870 (section 2).

A.4 Measurements results

N-methylaniline content X, %, is calculated by the formula (A.1):

$$X = 100 - \left(\sum Y_i + Z\right), \qquad (A.1)$$

where $\sum Y_i$ – sum of aniline, N,N-dimethylaniline and non-identified impurities contents, % (defined based on supplement B);

Z – water content, % (defined based on supplement B or GOST 14870 (section 2);

A.5 Presentation of measurements results

Measurements result in documents, provided its application, is represented as (A.2)

$$\left(X \pm 0{,}01\delta\right)\% \text{ at } P = 0{,}95 \qquad (A.2)$$

where X – N-methylaniline content, %;

δ – relative value of accuracy factor (see table A.1), %.

Measurement result is rounded up to one decimal place.

Supplement Б
(mandatory)

Determination of organic impurities content

А.1 Assigned characteristics of bias and measurements range

Assigned characteristics of bias method for probability level P = 0,95 and measurements range are given in the table В.1.

Table Б.1

Index	Measurements range, %	Relative value		
		Accuracy factor ±δ, %	Repeatability Limit $r_{отн.}$, %, n = 2	Reproducibility Limit $R_{отн.}$, %, m = 2
Aniline content	0,10 - 1,00 inclusive	22	17	29
N,N-dimethylaniline content	0,10 - 5,00 inclusive	22	9	31

2 Summary of test method

Method is based on gas chromatographic separation of organic impurities and main component over a packed column, their recording by means of a flame-ionization detector and results processing by internal standard method.

Б.3 Measuring devices, additional equipment, materials and reagents

В.3.1 *Measuring devices*

Gas chromatograph of type «Colorт 500M» with a flame-ionization detector and root-mean-square error of output signal ± 1 %.

Laboratory balance, GOST R 53228, accuracy class II, capacity 210 g, verification scale division 0,5 g.

Laboratory balance, GOST R 53228, accuracy class III, capacity 500 g, verification scale division 0,1 g.

Set (1 g-100 g) F_1 GOST OIML R 111-1.

Rule 300 GOST 427.

Lens LI 3-10 GOST 25706.

Microsyringe of 10 mcl.

Cylinder 1-10-2 GOST 1770.

Stop watch mechanical according to TC 25-1894-003.

Б.3.2 *Additional equipment*

Hydrogen generator producing hydrogen with volume fraction of main substance on a dry basis 99,99%, not less.

Glass or metal chromatographic column, length 2(3) m, internal diameter 2(3) mm.

Water bath ensuring temperature maintenance (80±2) °C.

Evaporation bowl 5 GOST 9147.

Glass СВ-24/10 GOST 25336.

Serum vial with a plug, volume 10 cm^3.

Glasses В-1(2)-100(250) ТХС GOST 25336.

Dropping vial 2-25 ХС GOST 25336.

Б.3.3 *Reagents and materials*

Aniline technical, STO 00204168-006-2009.

Compressed air to power control and measuring devices

Internal standard: benzyl alcohol, pure, GOST 8751.

Gas-carrier: technical gaseous nitrogen, GOST 9293.

N, N-dimethylaniline, pure, GOST 5855.

Ethyl alcohol, rectified, technical, best quality, GOST 18300.

Chloroform, chemically pure, GOST 20015.

Liquid stationary phase: apiezon L, production of Germany.

Solid support: chromaton N-AW-DMCS, fraction from 0,16 up to 0,25 mm.

Б.3.1 – Б.3.3 (Revised, Amendment № 1).

Б.4 Preparation for measurement

Б.4.1 Chromatograph mounting, adjustment and put into operation is carried out according to operation manual.

Б.4.2 *Preparation of packing*

15,0 g of chromaton N-AW-DMCS is placed to the evaporation bowl, KOH solution (0,15 g in 80 cm^3 of methanol) is added, mixture is heated in water bath at 70 °C - 80 °C with constant stirring till complete methanol removal. Then the packing is filled by apiezon L solution (2,5 g of apiezon L in 80 cm^3 of chloroform) and placed to evaporation bowl with solid support. Chloroform is removed by such way as methanol until the packing becomes loose.

N o t e – it's possible to apply the packing of chromatograph column without previous treatment of chromaton N-AW-DMCS by alkali. Analysis conditions has to ensure sample component separation similar to ones stipulated on figure 1.

(Revised, Amendment № 1).

Б.4.3 *Preparation of column*

Column is filled by the prepared packing, placed to a thermostat of chromatograph without connection to detector, purged by nitrogen current for 3-5 h at 170 °C. Then the column is cooled, connected to a detector and the chromatograph puts into operation.

Б.4.4 *Setting of calibration parameters*

Operating conditions of chromatograph

Temperature of columns thermostat, °C…… 130 -150

Temperature of evaporator, °C ……..230

Gas-carrier velocity, cm^3/min…… …… 30-40

Hydrogen flow velocity, cm^3/min…… ……..30-40

Air flow velocity, cm^3/min…….. ……..300-350

Movement velocity of diagram strip, mm/h……..……240

Sample volume, mcl……..……1-2

Setting of calibration parameters is carried out on three or more synthetic mixtures.

Synthetic mixtures are prepared as follows.

Weighted amount of 0,0050-0,3200 g of aniline, N,N-dimethylaniline and benzyl alcohol (internal standard) is placed to a glass for weighting (or serum vial) and diluted in 8 cm^3 of ethyl alcohol.

N o t e – if N-methylaniline without several impurities is available, calibration mixtures are prepared on its basis.

Each calibration mixture is chromatographed for three times, not less.

Peaks areas in all obtained chromatograms are calculated as the product of peak height by its width measured of the middle of height using a lens.

Calibration factor K_i of determined component is calculated by the formula (Б.1)

$$K_i = \frac{S_{эт} \cdot m_i}{S_i \cdot m_{эт}}, \qquad (Б.1)$$

where $S_{эт}$ – peak area of internal standard, mm²;

m_i – weighted amount mass of determined component, g;

$m_{эт}$ – weighted amount mass of internal standard, g;

S_i – peak area of determined impurity, mm².

Calibration factor of determined impurity is calculated as results average of all determinations.

Calibration factor of unidentified impurities is taken equal to the factor of the nearest known component.

Setting of calibration parameters is carried out once per a quarter, not less, as well as during the packing replacement and conditions change of chromatographic determination.

Stability check of calibration parameters is carried out once per a quarter, not less, according to recommendations of R 50.2.028-2003.

(Revised, Amendment № 1).

Б.5 Measurements

Weighted amounts of 0,0200 - 0,0400 g of benzyl alcohol and 2,0000 - 3,0000 g of analyzed N-methylaniline are placed to a glass for weighting. Mixture is stirred and fed to the chromatograph evaporator by a microsyringe.

Operation conditions of chromatograph are kept for measurements fulfillment according to point Б.4.4.

Chromatogram is registered and peaks areas are calculated.

Two parallel determinations are performed.

Order of components yield is indicated on the chromatogram (fig. 1).

Б.6 Measurements results

Content of determined component (aniline, N,N-dimethylaniline, unidentified impurity) Y_i, %, is calculated by the formula (Б.2):

$$Y_i = \frac{K_i \cdot S_i \cdot m_{эт.} \cdot 100}{S_{эт.} \cdot m_{пр}}, \qquad (Б.2)$$

where K_i - calibration factor of determined component;

S_i - peak area of determined component, mm²;

$S_{эт}$ - peak area of internal standard, mm²;

$m_{эт}$ - weighted amount mass of internal standard, g;

$m_{пр}$ - weighted amount mass of analyzed sample, g

Б.7 Acceptability test of parallel determinations results

Б.7.1 Results average of two parallel determinations is considered as measurements results provided that the following condition is performed (Б.3)

$$\left|Y_{i1} - Y_{i2}\right| \le r, \qquad (Б.3)$$

where Y_{i1}, Y_{i2} – results of parallel determinations of determined component, %;

r – absolute value of repeatability limit, %, calculated by the formula (Б.4)

$$r = 0{,}01 r_{отн} \frac{Y_{i1} + Y_{i2}}{2}, \qquad (Б.4)$$

where $r_{отн}$ - relative value of repeatability limits, %, indicated in table Б.1.

When the above condition is not performed, over limits causes are identified and eliminated and after that measurements are repeated.

Б.7.2 *Presentation of measurements results*

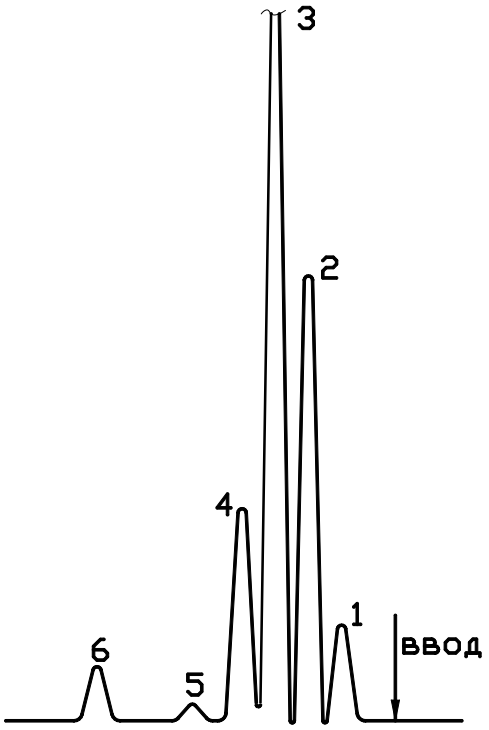
Measurements result in documents, provided its application, is represented as follows (Б.5)

$$\left(\overline{Y}_i \pm 0{,}01\delta\overline{Y}_i\right) \text{ \% at P = 0,95} \qquad (Б.5)$$

where $\overline{Y}_i$ – – results average of parallel determinations of determined component content (aniline, N,N-dimethylaniline) considered as acceptable %.

δ - relative value of accuracy factor of determined component (see table Б.1), %.

Measurement result is rounded up to the 2nd decimal.



1 – aniline; 2 – benzyl alcohol; 3 – N - methylaniline;
4 – N,N-dimethylaniline; 5,6 – unidentified impurities

Fig.1

Supplement B
(mandatory)

Determination of organic impurities content

A.1 Assigned characteristics of bias and measurements range

Assigned characteristics of bias method for probability level P = 0,95 and measurements range are given in the table B.1.

Table B.1

Index	Measurements range, %	Relative value		
		Accuracy factor ±δ, %	Repeatability Limit $r_{отн.}$, %, n = 2	Reproducibility Limit $R_{отн.}$, %, m = 2
Water content	0,05 - 0,50 inclusive	35	26	40

B.2 Summary of test method

Method is based on gas chromatographic separation of water and other sample components over a packed column, their recording by means of a thermal conductivity detector and results processing by internal standard method.

B.3 Measuring devices, additional equipment, materials and reagents

B.3.1 *Measuring devices*

Gas chromatograph of type «Colorт 500M» with a thermal conductivity detector and root-mean-square error of output signal ± 2 %.

Laboratory balance, GOST R 53228, accuracy class II, capacity 210 g, verification scale division 0,5 g.

Set (1 g-100 g) F_1 GOST OIML R 111-1.

Rule 300 GOST 427.

Lens LI 3-10 GOST 25706.

Microsyringe of 10 mcl.

Pipette 1-2-2-5 GOST 29227

Stop watch mechanical according to TC 25-1894-003.

B.3.2 *Additional equipment*

Glass or metal chromatographic column, length 2(3) m, internal diameter 2(3) mm.

Glass CB-24/10 GOST 25336.

Serum vial with a cup, volume 10 cm^3.

B.3.1, B.3.2 (Revised, Amendment № 1).

B.3.3 *Reagents and materials*

Distilled water GOST 6709..

Compressed air to power control and measuring devices

Internal standard: isopropyl alcohol, assay 99,8%, not less.

Gas-carrier: helium gaseous, volume fraction 99,995%, not less.

Packing: polysorb-1, fraction 0,25-0,50 mm under ND.

B.4 Preparation for measurement

B.4.1 Chromatograph mounting, adjustment and put into operation is carried out according to operation manual.

Б.4.3 *Preparation of column*

Column is filled by the packing, placed to a thermostat of chromatograph without connection to a detector, allowed to equilibrate by gas-carrier current for 5 h at 230 °C. Then the column is cooled, connected to a detector and the chromatograph puts into operation.

B.4.3 *Setting of calibration parameters*

Operating conditions of chromatograph

Initial temperature of columns thermostat, °C……....80-90
Final temperature of columns thermostat, °C…..…….230
Temperature of evaporator, °C ……....220
Velocity of temperature change of columns thermostat, °C/min……..…12-15
Gas-carrier velocity, cm^3/min ……35-45
Movement velocity of diagram strip, mm/h……..……600
Hold-up time of final temperature of column thermostat, s……600
Current of detector bridge, мА……100-120

Sample volume, mcl ……..……..……..1-3

Setting of calibration parameters is carried out on three or more synthetic mixtures with water content 0,05-0,50%.

Synthetic mixtures are prepared as follows.

Weighted amounts of 0,0050-0,0250 g of water and 0,0050-0,0500 g of isopropyl alcohol are placed to a glass for weighting (or serum vial) and diluted in 5,0 cm^3 of N-methylaniline with water content 0,05%, not more.

Each calibration mixture and initial N-methylaniline are chromatographed for three times, not less.

Peaks areas in all obtained chromatograms are calculated as the product of peak height by its width measured of the middle of height using a lens.

Water calibration factor $K_в$ is calculated by the formula (B.1)

$$K_в = \frac{S_{эт} \cdot m_в}{\left(S_в - S_0\right) \cdot m_{эт}}, \qquad \text{(B.1)}$$

where $S_{эт}$ – peak area of internal standard, mm^2;

$m_в$ – weighted amount mass of water, g;

$m_{эт}$ – weighted amount mass of internal standard, g;

$S_в$ – peak area of water in synthetic mixture, mm^2;

S_0 - average peak area of water in initial N-methylaniline, mm^2.

Factors obtained over calibration mixtures are averaged.

Setting of calibration parameters is carried out once per a quarter, not less, as well as during the packing replacement and conditions change of chromatographic determination.

(Revised, Amendment № 1).

B.5 Measurements

Weighted amount of 3,0000 - 5,0000 g of N-methylaniline is placed to a serum vial keeping closed a rubber plug, 0,0100 - 0,0300 g of isopropyl alcohol is added by a microsirynge. Mixture is carefully stirred and placed to chromatograph evaporator.

Operation conditions of chromatograph are kept for measurements fulfillment according to point Б.4.3.

Chromatogram is registered and peaks areas are calculated.

Two parallel determinations are performed.

Order of components yield is indicated on the chromatogram (fig. 2).

B.6 Measurements results

Water content Z, %, is calculated by the formula (B.2)

$$Z = \frac{K_в \cdot S_в \cdot m_{эт.} \cdot 100}{S_{эт.} \cdot m_{пр}}, \qquad (B.2)$$

where $K_в$ - calibration factor of water;

$S_в$ - peak area of water in analyzed sample, mm^2;

$S_{эт}$ - peak area of internal standard, mm^2;

$m_{эт}$ - weighted amount mass of internal standard, g;

$m_{пр}$ - weighted amount mass of analyzed sample, g

B.7 Acceptability test of parallel determinations results

B.7.1 Results average of two parallel determinations is considered as measurements results provided that the following condition is performed (B.3)

$$\left|Z_1 - Z_2\right| \le r, \qquad (B.3)$$

where Z_1, Z_2 – results of parallel determinations, %;

r – absolute value of repeatability limit, %, calculated by the formula (B.4)

$$r = 0{,}01 r_{отн} \frac{Z_1 + Z_2}{2}, \qquad (B.4)$$

where $r_{отн}$ - absolute value of repeatability limit, %, (see tablr B.1).

When the above condition is not performed, over limits causes are identified and eliminated and after that measurements are repeated.

B.7.2 *Presentation of measurements results*

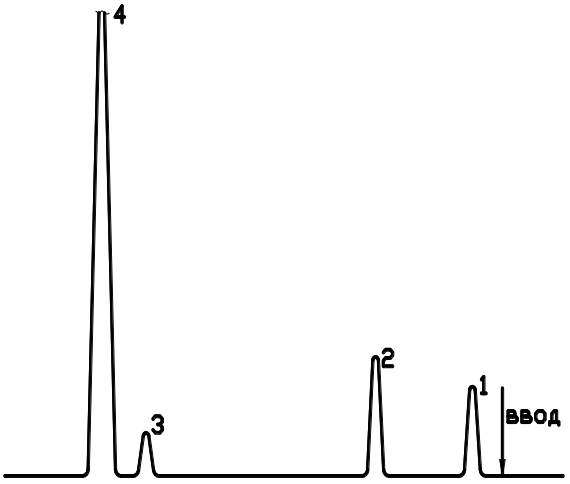
Measurements result in documents, provided its application, is represented as follows (B.5)

$$\left(\overline{Z} \pm 0{,}01\delta\overline{Z}\right) \text{ \% at P = 0,95} \qquad (B.5)$$

where $\overline{Z}$ – results average of parallel determinations of water content considered as acceptable %.

δ - relative value of accuracy factor,%, (see table B.1).

Measurement result is rounded up to the 2nd decimal.



1 – water; 2 – isopropyl alcohol; 3 – aniline;
4 – (N- methylaniline + N,N-dimethylaniline)

Fig. 2

Supplement Г
(mandatory)

Determination of color

Г.1 Summary of test method

Measuring method of N-methylaniline color is based on photometric definition of optical density at light wave length (540±20) nm.

Г.2 Measuring devices

Photoelectrical photometer having cuvettes with working length l = 5 mm and admissible value limit of main absolute photometer error at transmittance factor measurement 0,5 % abs., not more (type KFK-3 according to TC 3-3.2164-89).

фотоэлектрический имеющий кюветы с рабочей длиной мм и предел допускаемого значения основной абсолютной погрешности фотометра при измерении коэффициента пропускания не более 0,5 % абс. (типа КФК-3 по ТУ 3-3.2164-89).

Г.3 Measurements

Analyzed product is poured to a cuvette with working length l = 5 mm and its optical density is measured in comparison with distilled water at wave length λ = (540 ± 20) nm.

Optical density measurement is carried out twice, not less.

Measurement result is recorded up to the 3rd decimal.

Г.4 Measurements results

Results average of two parallel determinations is considered as measurements results, discrepancy between measurements has not to exceed 0,002.

(Revised, Amendment № 1).

Supplement Д
(reference)

Normative and other references*

ГОСТ Р 1.4-2004	Standardization in the Russian Federation. Standards of organizations. General provisions.
ГОСТ Р 53228-2008	Automatic balance. Part 1. Metrological and technical requirements. Verification.
ГОСТ Р ИСО 5725-6-2002	Accuracy (trueness and precision) of measurement methods and results.
ГОСТ 12.1.007-76	SSBT. Hazardous substances. Classification and general safety requirements.
ГОСТ 313-77	Aniline technical. Specifications.
ГОСТ 427-75	Metal measuring rules.
ГОСТ 511-82	Fuel for engines. Motor method for octane number definition.
ГОСТ 1770-74	Glass lab volumetric ware. Cylinders, beakers, flasks, tubes. General specifications.
ГОСТ 2517-85	Petroleum and its products. Sampling methods.
ГОСТ 5855-78	N,N-dimethylaniline. Specifications.
ГОСТ 6709-72	Distilled water. Specifications.
ГОСТ 6732.2 -89	Organic dyes. Intermediates for dyes, textile auxiliaries. Sampling methods.
ГОСТ OIML R 111-1-2009	GSI. Weights of accuracy class E (index 1), E (index 2), F (index 1), F (index 2), M (index 1), M (index 1-2), M (index 2), M (index 2-3), M (index 3). Part 1. Metrological and technical requirements.
ГОСТ 8751-72	Benzyl alcohol. Specifications.
ГОСТ 9147-80	Porcelain lab ware and equipment. Specifications.
ГОСТ 9293-74	Gaseous and liquid nitrogen. Specifications.
ГОСТ 14192-96	Labeling of goods.
ГОСТ 14870-77	Chemical goods. Water determination methods.
ГОСТ 15846-2002	Products delivered to regions of the Far North and equated localities.
ГОСТ 18300-87	Ethyl alcohol, rectified, technical. Specifications.
ГОСТ 19433-88	Hazardous goods. Classification and labeling.
ГОСТ 20015-88	Chloroform. Specifications.
ГОСТ 25336-82	Glass lab ware and equipment. Types, main parameters and dimensions.
ГОСТ 25706-83	Lenses. Types, main parameters. General specifications.
ГОСТ 26380-84	Special composite containers. Types, main parameters and dimensions.
ГОСТ 30302-95 (ГОСТ Р 50610-93)	Special containers. Types, main parameters and dimensions.
ГОСТ 31314.3-2006 (ИСО 1496-3:1995)	Freight cars of series 1. Technical requirements and test methods. Part 3. Tanks-containers under pressure for liquids, gases and bulk cargo.
МИ 2335-2003	Internal quality control of quantitative chemical analysis results.
Р 50.2.028-2003	Algorithms of calibration parameters definition of measuring devices for substances and materials composition and evaluation of their errors (uncertainties). Error (uncertainty) evaluation of linear calibration parameters using least squares method.
РМГ 76-2004	Internal quality control of quantitative chemical analysis results.
ТУ 25-1894.003	Stop watch mechanical. Specifications.

***** The above normative and reference documents were valid at the moment of STO approval. In future to apply the present standard it's reasonable to check the actuality of reference standards in public information system – in Internet on the official site of the Federal Technical Regulation and Metrology Agency or in annual information index "National standards" published as per January 1 of the current year and according to appropriate monthly reference index published in the current year. In case of replacement (amendment) of the reference document, to apply the present standard it's necessary to follow the replaced (amended) standard. In case of the document cancellation without replacement, then the provision with the reference to it is applied in the part being without prejudice to this reference.

(Revised, Amendment № 1).

Abbreviations, terms and definitions

IUPAC name – International Union of Pure and Applied Chemistry name, recognized by international scientific community.
CAS – Chemical Abstract Service Registry Number, recognized by international scientific community.
RTECS – Toxicological Effects of Chemical Substances Registry number, recognized by international scientific community.
EINECS –control number in the European Inventory of Existing Commercial Chemical Substancesв.
UN/OOH – four-digit identification number of substance or article in UN Recommendations on the Transport of Dangerous Goods (Standard rules).
ICSC CARD - International Chemical Safety Card

Shelf life: Time period, within which the manufacturer ensure all standard consumer properties of the product provided that storage conditions are kept. Upon expiry of shelf life, usually the product loses its consumer properties.
Guarantee shelf life: Time period, within which the manufacturer ensure all standard consumer properties of the product provided that storage conditions are kept and the buyer may present a claim in case of any product defect.

Bibliography

[1] ГН 2.2.5.1313-03	Chemical factors of production environment. Extreme admissible concentration (EAC) of harmful substances in working zone. Hygienic standards (as amended by the Addendum No. 7 approved by the Resolution of Chief State Sanitary Doctor of the Russian Federation No. 96 of 12.07.2011).
[2] ГН 2.1.6.1338-03	Extreme admissible concentration (EAC) of contaminants in ambient air of inhabited areas. Hygienic standards (as amended by Resolution of Chief State Sanitary Doctor of the Russian Federation No. 26 of 03.11.2005, amendment of 12.07.2011).
[3] ГН 2.1.5.1315-03	Extreme admissible concentration (EAC) of chemical substances in water of aquatic reservoirs of drinking water and consumer application. Hygienic standards (as amended by Resolution of Chief State Sanitary Doctor of the Russian Federation No. 77 of 28.09.2007).
[4] Rules for Transportation of Liquid Goods in bulk in tanks-cars and in hopper wagons for transportation of oil bitumen	Approved by the European Rail Council of countries – EC members (protocol № 50 of May 21-22, 2009).
[5] Regulations concerning the International Carriage of Dangerous Goods by Rail	Approved by the European Rail Council of countries – EC members, Protocol № 15 of 05.04.1996 (as revised and amended on 23.11.07, 30.05.08, 22.05.09; as amended by protocols of 14.05.2010, 21.10.2010, 29.10.2011)
[6] Technical requirements of the Goods placement and fastening in wagons and containers	Approved by the order of the Ministry of Railways of the Russian Federation No. ЦМ-943 of 27.05.2003 (as amended by letter of JSC “Russian Railways” No. 139 of 12.07.2004, amendments No. ЦМУ-6/279 of 12.08.2005).
[7] Regulations concerning the International Carriage of Dangerous Goods to the Agreement on International Goods Transport by Rail (SMGS).	Supplement 2. Organization of railways cooperation (ОСЖД) (as amended of 01.07.2009).
[8] The Regulations concerning the International Carriage of Dangerous Goods by Road”	Approved by the Government Resolution of the Russian Federation No. 272 of 15.04.2011 (as amended by the Government Resolution of the Russian Federation No. 1208 of 30.12.2011).
[9] ECE/TRANS/215	European Agreement on International Goods Transport by Trucks (DOPOG). Volumes 1,2. Published by UN, 2010.

(Revised, Amendment № 1).