OLIGOETHERACRYLATE (of grade MTQ-9)

SPECIFICATIONS TY 6-01-450-76

The present specifications cover oligoetheracrylate of grade MTQ-9.

Oligo ether acrylate is transparent liquid with weak smell of toluene.

Oligo ether acrylate MTQ-9 is obtained by method of condensing polymerization from/ methacrylic acid, triethylene glycol and phthalic anhydride.

Oligo ether acrylate MTQ-9 is used as a binder in production of reinforced plastics, protecting coatings, constructions material, electric insulation compounds, lacquer, special pastes and others compounds. X

Example of conditional designations for ordering oligoetheracrylate of grade MTQ-9 :- OEAMTQTY-6-01-450-76.

Oligo etheracrylate MTQ-9 is combustible easy polymerizing liquid with density in 20°C within the range of 1.14-1.18 g/cm³ insoluble in water, good soluble in organic solvents, acetone, benzole, toluene, chloroforms.

The small quantity of toluene is available in commercial product which is easy volatile matter. the limit acceptable concentration shall be taken by toluene solvent.

1. Technical requirements : Oligo ether acrylate shall comply with the present technical requirements .

1.1. Oligo ether acrylate MTQ-9 is produced of two grades, high quality and first quality instead of grade MTQ,-9.

1.2. Oligo ether acrylate MTQ-9 is manufactured in accordance with process chart developed in established order.

1.3. Main characteristics (Properties) - Oligo ether acrylate MTQ-9 shall comply with requirement and norms by physical and chemical properties noted in the table

Name of indicates	Norms for grades X		Test procedure
	High	First	
General appearance	Transparent Liquid having Colour between yellow and dark brown.		As per 4.1 issue of the present specification.
by brookfield. Viscosity in Temp. 20± 0.25°C in Cp.	100- 200	100- 250	As per TOCT 33-66 and 4.2 issue of the specification.
Content of main substance in % not less	97	96	As per 4.3 issue of the specification.
Content of Toluene in % not to exceed	3	4	As per 4.4 issue of the specification.

Certified for use

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[illegible]ntent of hydro [illegible]inone in %	0.030-0.070	0.030-0.075	as per 4.5 issue of the specification.
Content of water [illegible] not to exceed	0.1	is not determined	as per TOCT 148 to 69 and 4.6 issue of the specification
[illegible] not to exceed	0.04	0.04	as per 4.7 issue of the specification
Saponification number mg KOH [illegible] ☆ [illegible] not ~~be~~ less than exceeding	384	380	as per 4.8 issue of the specification.
Acid number in the moment of manufacturing mg KOH [illegible] not be exceeding	1.0	5.0	as per 4.9 issue of the specification
[illegible]lymerization rate in 100°C temp. with 1% benzoyl peroxide in minutes.	is not determined	1.3	as per 4.10 issue of the specification
Polymerization activity in minutes not less than	40	30	as per 4.11 issue of the specification.

☆ not less than 384

3.3. Sampling

The average sample is selected from every lot for conducting of tests.

3.3.1. Sampling from barrels and bottles is performed with the help of glass pipe. Dipping of glass pipe in liquid is performed slowly with opened upper end so that sample would be uniform by hight height. Sample is taken from barrels from 10% of packaged places but not less from three places of small lot.

3.3.2. One averaged sample is selected from tank. Sampling from tankcars or containers is performed with sampler from three layers of liquid.

3.3.3. Quantity of sample shall be not less than 1 liter.

3.3.4. One half sample is transfered for test and another is remainded as a sample for reference.

3.3.5. Storage of samples for reference is performed during 6 months.

3.3.6. Labortory for carrying out of analusis is selected on agreement of sides.

If analysis results are unsatisfactory, the second analysis of sample from doubled quantify of places is carried out by indication in compliance with the present specifications requirement only.

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4. TESTS PROCEDURE.

4.1. Vessels for use:-

Test glasses as per TOCT 10515-73 of type nx12+1, 14+1 mm.

4.1.2 Execution of test:-

Estimation of general appearance is performed by visual inspection of oligoether acrylate sample by transmitted liquid in 50-60 mm. height of product layer.

4.2. Determination of viscosity:-

Viscosity is determind in accordance with TOCT-33-66 in tempe temperature 20+025°c. Viscometer as per TOCT-10028-67 is used for this purpose, the type B II *-1 with capillary diameter dk-2.1± 0.04mm.

The calculations is conducted by formula.

n - K.dr

Where n - viscosity in cp

K - Viscometer constant

d - oligoether acrylate density equals to 1.16g/

T - efflux time in second.

4.3. Determination of content of main substance:-

Percentage is determind by formula

$$X = 100 - X_{np}$$

Where Xnp is admixtures content, toluene, hydroquinone etc. It is permitted to determine content of main substance by saponification number calculation is conducted by formula.

% main substance = $X = \frac{X3}{X_0} \cdot 100$ X3 observed saponification number; X_0 — theoretical

Where X3 is practice saponification number for oligoether-acrylate MTQ-9 determinedin accordance with 4.8 issue of the specification. X_0 is theoretical saponification number for oligoether acrylate MTQ-9 equal to 395.7.

Umpire method is by difference (100-admixture)

4.4. Determination of toluene content.

Determination of toluene content in oligoether acrylate MTQ-9 is performed by gas- liquid distributing chromatography with using of catarometer.

4.4.1. Used equipments and reagints:-

Gas chromato graph with detector by thermoconductivity(U, bel

Injector for 1 & 2 ml. (type record)

Flasks with self sealing rubber corks 100 ml.capacity.

Spherochrome-1 (THD-TC-M) grains are 0.32-0.50mm.

Standard solution, Benzene as per TOCT 6955-70 + Chlorobenzene as per TOCT 3486-68 (1:10)

oligoetheracrylate without toluene.

The standered column of chromatograph with inner diameter 4mm and lenth 1m is used for conducting of analysis solvent is [illegible]-1 (TND-TC-M) with graining of 0.32-0.50mm. containing immovable phase in Sty-10-15% from carrier gas.

Preparation of solvent:-

Weight portions of carrier is impregnated by immovable phase solution in petroleum ether and dried up to loose condition on the water bath.

Starting of device and conducting of analysis:-

Starting of device is performed in compliance with device manual operating temperature of thermostat is established 100°c gas-carrier consumption -7.5 l/h. After establishing the zero line the sample of vapours which are available above oligoetheracrylate are introduced by injector. The injector required to blow thoroughly after every charging.

Quantity analysis to be done by standered additive:-

Several samples of polyether with specified 1,2,3,5,7,10 % toluene contents are prepared for constructing the curve, weight portion is near 4 g. Then standered solution in quantity of 1% from polyether with toluene is poured in flasks. The content of flask is stirred thoroughly and heated on boiled water bath. Vapour sample is taken by injector and introduced in column 1.2ml. Then chromatographic peack of benzene, toluene, chlorobenzene are measured and the values are substituted in expression:-

$$\frac{ht\% \text{ standard solution}}{hb + hob.\ X}$$

Where ht, hb, hob -- are the hights of correspondent peacks of toluene, benzene, chlorobenzene in mm is calibration coefficient, practically determined.

The calibration curve is constructed by obtained data.

Value of $\frac{ht\% \text{ standard solution}}{hb + hob \cdots X}$ is layed off as ordinate and toluene content in % as obtained curve is employed for determining of toluene content in oligoether acrylate.

4.5. Determination of calibration coefficient(X)

5 g of toluene is wighed with accurecy 0.0002 g. with analitical balance and equal quantity of chlorobenzene and benzene (5-10% of toluene weight) are added mixture is stirred and introduced with microinjector in chromatograph column is quantity of 1ml. Then height of chromatograph peaks of benzene and chlorobenzene are measured and taken thir relation.

$$X = \frac{H \text{ benzene}}{H \text{chlorobenzene}}$$

It is recommended to prepare 2-3 mixtures of toluene-benzene chlorobenzene and make 3-5 chromatographes with every mixture. Calibration coefficient "X" is found as arithmetic means of received values.

4.5. ~~Determkkkxxxxfx~~ Determination of hydroquinone content:-

The method based on reaction of hydroquinone with phloroglucinol in weak alkali medium.

4.5.1 Used reagents, solutions devices, vessels.

Caustic soda as ГОСТ 4328-66, 0.5 N solution.

Toluene analytically pure as per ГОСТ 5789-69.

Methanol as per ГОСТ 2222-70.

Mixture of toluene methanol 2:3.

NA Phloroglucinol 0.3% water solution, fresh prepared.

NA Photo electric colorimeter.

Volumetric flasks as per ГОСТ- 1770-74 having 50, 100,200ml.cap

Pipettes as per ГОСТ 20292-71 having 1,10 15,25ml.capacity.

4.5.2 Preparation of standard mixture:- 0.0200grms. of fresh distilled hydroquinone is weighed with analytical balance, dissolved in toluene- methanol mixture in 200ml.volumetric flask.stirred thoroughly. 1ml. of solution contains 0.1mg. of hydroquinone.

4.5.3. Construction of calibration curve:- The following quantity of hydroquinone standard solution measured by microburette.

0.1ml - 0.01mg, 1.0ml-0.1mg, 1.50ml-0.150mg & so on, are placed in 50ml. volumetric flasks. then toluene methanol mixture is added and stirred after that ~~xxSNxNaOHxxsolutionxxxx~~ 0.2ml. of fresh prepared phloroglucinol solution and 1.0ml. 0.5N NaOH solution are poured in flask content is stirred and led to mark by toluene methanol mixture. Then it stirred thoroughly and measured optical density with photo colorimeter through 40 min. after pouring in. Measurement of optical density is performed with respect to toluene methanol mixture with blue filter No.3. and 20mm. thickness of layer in cell on the left barrel.

Have measured optical density the calibration curve is constructed by laying off optical density as ordinate & hydroquinone concentration as abscissa. Calibration curve has the appearance of straight line cutting by ordinate axis the value equal to optical density of standard solution prepared out of 48.8ml. toluene-methanol mixture, 0.2ml. fresh prepared phloroglucinol solution and 1.0ml.of 0.5N NaOH solution.

4.5.4. Description of determination:- 2.4-3.6g weight portion (in dependence on expected content of hydroquinone in O.E.A.) is dissolved in toluene methanol mixture(2:3) in 100ml. volumetric flask and 0.2ml. of fresh prepared phloroglucinol solution and 1ml of 0.5N NaOH solution are poured in, flask cont content is stirred and led to the mark with solvent mixture. Then it stirred thoroughly and through 40 min. optical density is measured as described in issue pertaining to calibration curve construction.

The optical density of working solutions is determined for introducing correction for colour, for this purpose 10 ml. solution is taken from 100ml. flask from solution of the same weight portion and placed in 50ml volumetric flask, then volume of solution is led to the mark with toluene methanol -

Opticalum density measurement is performed as well as the left barrel with blue filter from optical density of working solution with phloroglucinol. The optical density of solvent substracted then hydroquinone content is found by calibration curve.

Hydroquinone content in % is calculated by formula:-

$$X_1 = \frac{m_1 \times 100 \times 100}{m \times 1000 \times V}$$

where m_1 is hydroquinone content found by calibration curve in mg, m is weight portion of oligo ether acrylate, 1000 for conversion of mg to g, v is solution volume of weight portion taken for colorimetring in ml.

Determination of hydroquinone is conducted by two weight portions parallelly. Accuracy of technique is 10%.

4.6. Determination of water content:- Water content is determine in accordance with TOCT 14870-69 by fisher's method with electrometri titrations.

4.7. Determinations of SO_4^{--} ions contents.:- The essence of method is in dissolving of weight portion of oligoetheracrylate in methanol, precipitation of SO_4 ions by $BaCl_2$ solution and titration of Ba^{++} as was excess by Trilon B in presence of methyl thymol blue indicator.

4.7.1 Required reagents, solutions, vessels.

Trilon B as per TOCT 10652-73, 0.02N solution (3.72g of trilon B is dissolved in 1 ltr. of distilled water).

Barium chloride as per TOCT 4108-72, 0.02N solution (2.44g $BaCl_2 \cdot 2H_2O$ is dissolved in 1 ltr. of distilled water)

Buffer solution (pH-12.3) is prepared by mixing of two solutions A & B in relation 3:7. Solution A is 7.507g of glycol, 5.85g of NaCl per 1 ltr. of water. Solution B is 0.1N solution of NaOH.

Methyl thymol blue is indicator. Methanol. Magnesium sulphate. Conical flasks 100ml cap. Pipette max 5ml cap.

4.7.2 Determination procedure :- Weight portion of oligoetheracrylate 2g. approx. (weight portion to be taken from dropping bottle) is placed in 100ml flask, where 30ml. of methanol poured preliminari After dissolving 5ml of 0.02N $BaCl_2$ is added with pipette, stirr thoroughly and 10ml. of buffer solution poured in. The methyl thymol blue indicator is added up to clear colour and titrated w 0.02N solution of Trilon B up to its disappearance.

Checking tibration is conducted as follows:- 5ml. 0.02N barium chloride solution, buffer solution and methyl thymol blue indica is added to 30ml methanol then it is tibrated with 0.02N Trilon solution up to blue colour disappearance.

Determination of titre of 0.02N Trilon B solution as follows:- 10ml. 0.02N magnesium sulphate ($MgSO_4$) solution & 20ml of the distilled water and 5ml. of ammonia buffer solution(pH-10) are titrated with 0.02N Trilon B solution with chrome dark blue indi up to transition of wine red to blue solution colour.

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4.7.3. Data processing:- Content of SO_4 ions (x_2) in presents is calculated by formula:-

$$X_2 = \frac{(V_1 - V_2) \cdot K \cdot 0.00096 \times 100}{m}$$

Where V_1 - Quantity in ml. of 0.02N Trilon B taken for checking titration (Blank).

V_2 - Quantity in ml. of 0.02N Trilon B taken for working titration.

K - Correction of 0.02N Trilon B solution(Factor)

0.00096 - SO_4 ions quantity in gr. corresponding to 1ml. 0.02N Trilon B solution.

M - Weight portion in g. of oligoether acrylate.

4.8. Determination of saponifications number:-

4.8.1. Used vessels and reagents:-

Conical flask 250ml.capacity, Burette 50ml.cap., dropping bottle for weighing, cylinder with nose 5ml.cap.

Phenolphthalein 1% alcohol solution, isopropyl alcohol, Caustic potash or caustic soda 0.5N solution, hydrochloric acid or sulphuric acid 0.5N solution, distilled water.

4.8.2 Test execution:- 20ml of nutralized isopropyl alcohol is poured in 250ml. cap. flat bottom flask then 1-2g. weight portions of oligoetheracrylate weighed with the help of dropping bottle with accuracy up to 0.0002g. is placedin .Flask content is neutralized with 0.1N of caustic potash or caustic soda solution with indication by phenolphthalein then accurately 40ml of 0.5N caustic potash or soda water solution is poured in.

Flask is connected to reflux condenser and boiled on the closed electrical plate in 15 min.

Hot solution is titrated with 0.5N hydrochloric or sulphuric acid solution in the presence of phenolphthalein up to disappearing of pink colour. Reference sample without weight portion is put in the same conditions parallelly. (Blank)

4.8.3. Data processing:- Saponification number(X_3) is determined by formula.

$$X_3 = \frac{(V - V_1) K \times 0.02805 \times 1000}{m}$$

Where V - Volume of 0.5N sulphuric acid or hydrochloric acid solution consumed for reference sample titration in ml. (Blank)

V_1 - Volume of 0.5N sulphyric acid or hydrocholoric acid consumed for titration of sample under test in ml.

K - Correction coefficient for tire of 0.5N sulphuric or hydrochloric acid solution. (Factor)

0.02805 - Caustic potash quantity in g corresponding to 1ml of 0.5N sulphuric acid or hydrochloric acid solution.

m = Oligoetheracrylate mass in g.

4.9. Determination of acid number:-

4.9.1. Used vessels and reagents.

Conical flasks 100,250ml. cap.,microburette,Caustic soda or caustic potashwater solution 0.01N or 0.1N. Phenolphthalein 1% alcohol solution. Isopropyl alcohol, Distillwater.

4.9.2. Execution of test:- 2-3g. oligoetheracrylate weight portion each place in 100, 250ml cap.conical flask and dissolved in 20ml. of nutralized isopropyl alcohol. Then it is stirred thoroughly and titrated with 0.01N caustic soda or potash solution in phenolphthalein presence. Titration is carried out with microburette.

4.9.3 Data processing. - Acid number(X_4) is calculated by formula.

$$X_4 = \frac{V \cdot K \cdot 0.00056 \cdot 1000}{m}$$

Where V - quantity of 0.01N alkali solution consumed for weight portion titration in mg. ml

K - Correction coefficient for 0.01N alkali solution

0.00056 - Quantity of caustic potash corresponding to 1ml. 0.01 N alkali solution in gram.

m - Oligoetheracrylate mass in g.

4.9.4. In case of discrepancy of analysis out comes of saponification numbers and acid number between parallel. Samples of manufacturer's and consumers results caustic soda or potash waters solutions shall be replaced to caustic soda or potash alcohol solution of the same concentration.

4.10. Determination of rate of polymerization :-

4.10.1. Used vessels and reagents:-Conical flasks.100,250ml.capacity, test glasses,12,14mm.diameter,benzoyle peroxide industrial grade.

4.10.2 Test execution:-OLIGOETHERACRYLATE weight portion (10-30g.)is weighted in 100ml. capacity flask, then 1% of dry benzoylperoxide is added to it. then stirred thoroughly (30min. approx.)up to complete dissolutions.

Then portion of obtained composition is transfered in the test glass and placed it in the boiled water bath (liquid amount in atest glass should be 20-30min.) .Time since moment of testglass dipping in water to appearance of peeling off test glass walls separated polymer is measured with stop watch.

4.11 Determination of activity of polymeryzation:- Method of polymerization activity determination of oligoetheracrylate [illegible] is based on measuring oligoetheracrylate dielectric constant in polymerization process.

4.11.2 Used devices and reagents. Capacity transluser, contact thermometer 0-100°c, Unit of temperature adjestment, 50ml flask, industrial benzoyl peroxide .

4.11.3 Preparation for determination switching on DIOD-1, device and preparation of transluser is carried out in accordance with manual supplied with device.

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4.11.4. Preparation of mixture. :- Preparation of mixture is stated through 30 min. after transducer [illegible] warming up start. 10-15g. oligoetheracrylate MTG-9 weight portions is weighted with 0.01g accuracy in 50ml. flask. then dry benzoyl peroxide is added and mixture is stirred thoroughly in 30 min. up to complete dissolution of benzoyl peroxide (particle of benzoyl can not be seen.).

4.11.5. Polymerization process monitoring mixture of OEA [illegible] MTG-9 with benzol peroxide is filled in polymerization transducer chamber as a thin layer.(5-7ml.) then chamber is closed by cover and recorder pen is put on 95-98 divisions of diagram tape. After this the device of registers polymerization process in transducer automatically. Polymerization process is considered completed, recorder readings varies not more than 2 diagram divisions in the last 10 minutes.

4.11.6. Determination of polymerization activity :- ~~Number of divisions~~ Number of divisions Amax is accounted on diagramtape on which recorder pen has deflected from the begining of polymerization process. The point corresponding to recorder pen deflections on

$$A0.5 = \frac{A.max}{2}$$

divisions is found on oligoetheracrylate.

MTG-9 During kinetic curve. Time(T0.5) during which recorder pen deflected up to this point, taking for origin the moment of device adjustment with filled transducer is determined by diagram tape in minutes. Example of T0.5 that determination is given on the figure 1.

Time for T0.5 determinations including preparation of transducer, oligoetheracrylate MTG-9 with benzoyl peroxide mixture and warming up to device and thermostate depends on oligoetheracrylate MTG-9 lot and equals to 2-4 hours accuracy of determination of T0.5 time is 10% while T0.5-20-30 minutes.

5. TRANSPORTATIONS AND STORAGE.

5.1. Oligoetheracrylate transporation is carried out by any type of transport with compulsory pre-vention from sunlight.

5.2. Oligoetheracrylate storage is performed in storage premises without sunlight admittance in themperature -25°C to +25°C.

6. Manufacturer guarantees.

6.1. Oligoetheracrylate shall be accepted by manufacturer [illegible] technical inspection.

6.2. Manufacturer guarantees oligoetheracrylate compliance with the presence specifications requirements, If consumer observes condition of transportations and storage established with the present specification.

6.3. Guarantee is established for 6 months from the date of mfg.

6.4. After lapse of guarantee time before every use the product is analysed for compliance with the specifications requirement and may be used on designated purpose if results are satisfactory.

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