

KES

General Method of Alkyl Resin

KES T – M156

(EST.) 1989 – 02 – 28

Contents

1. Scope of Application
 2. Fatty Acid Content
 3. Glycerol, Ethylene Glycol
 4. Pentaery Thritol
 5. Isophthalic Acid
 6. Phthalic Acid Isomers and Benzoic Acid
 7. Carboxylic Acid
- Additional Rule



1. Scope of application

This standard shall specify the quantitative and qualitative analysis method of the whole ingredient in alkyd resin.

2. Fatty acid content

2-1 Scope of application

This standard shall specify the fatty acid content test method in alkyd resin.

2-2 Reagent

- (1) Ethyl ether
- (2) Sodium sulfate
- (3) Sulfuric acid solution (1 : 4)
- (4) Congo red paper
- (5) Methyl orange indicator: make 100 mL solution that melt 0.1 gr of methyl orange in water.

2-3 Test method

- (1) Remove filtrate according to phthalein acid in alkyd resin anhydride content test method into a 400 mL beaker, evaporate in a steam bath until filtrate is desiccated completely, and put 10 mL of water. After evaporating until the solution is dried again, eliminate all volatile solvent to have put a beaker into a 105 ± 2 °C desiccator.
- (2) Melt dregs in 100 ~ 150 mL of water, neutralize them with sulfuric acid solution for the use of congo red by indicator. You should prevent isolated fatty acid from contacting with air as putting 25 mL of ether instantly.

Remove the contents into 500 mL of a separatory funnel After washing the beaker with 25 mL of water, and put into the separatory funnel. You should operate as above using ether.

- (3) Add 25 mL of ether, and cover the lid of the separatory funnel. Shake contents slowly and fully.

Let alone some while. Remove a layer of water into different separatory funnel when contents divide two layers, and extract again by putting 50 mL of ether.

You should repeat the extraction using 25 mL of ether at a time until the layer of ether has been colorless.

- (4) Remove all of extract liquid into the first separatory funnel, and cleanse them with 25 mL of water at a time for the use of methyl orange by indicator until they are neutralized.

At this time, be careful, since if the funnel is shaken severely, the ether layer will be in a lactated state and will not be separated.

- (5) Remove ether solution a 300 mL Erlenmeyer flask including 15 ~ 25 gr of absolute sodium sulfate, cleanse the separatory funnel with a little ether, and put it.

Cover the lid of the flask, and shake until the solution is cleared. After filtering a 125 mL Erlenmeyer flask that is aware of the weight, evaporate it in a steam bath.

Cleanse sodium sulfate, the flask, and the filter with ether. Put them into the above flask, and evaporate again. When most of ether is evaporated, evaporate completely as passing through dried carbon dioxide.

- (6) Unless the flask smells ether, put it into a 105 ± 2 °C desiccator, and dry about 10 minutes as passing through carbon dioxide.

Put the flask into a vacuum desiccator including sulfuric acid, dry continuously until content is satisfied, and weigh it.

If you need, measure a refractive index, iodine number, and etc. When you don't use, store in carbon dioxide air current or a vacuum desiccator.

(7) Calculate a percentage of fatty acid content in the specimen as the following formula.

$$\text{Fatty acid content (\%)} = \frac{A}{S} \times 100$$

A : Weight of precipitated fatty acid

B : Weight of inert material or solid color material in the specimen

3. Glycerol, Ethylene Glycol

3-1 Summary

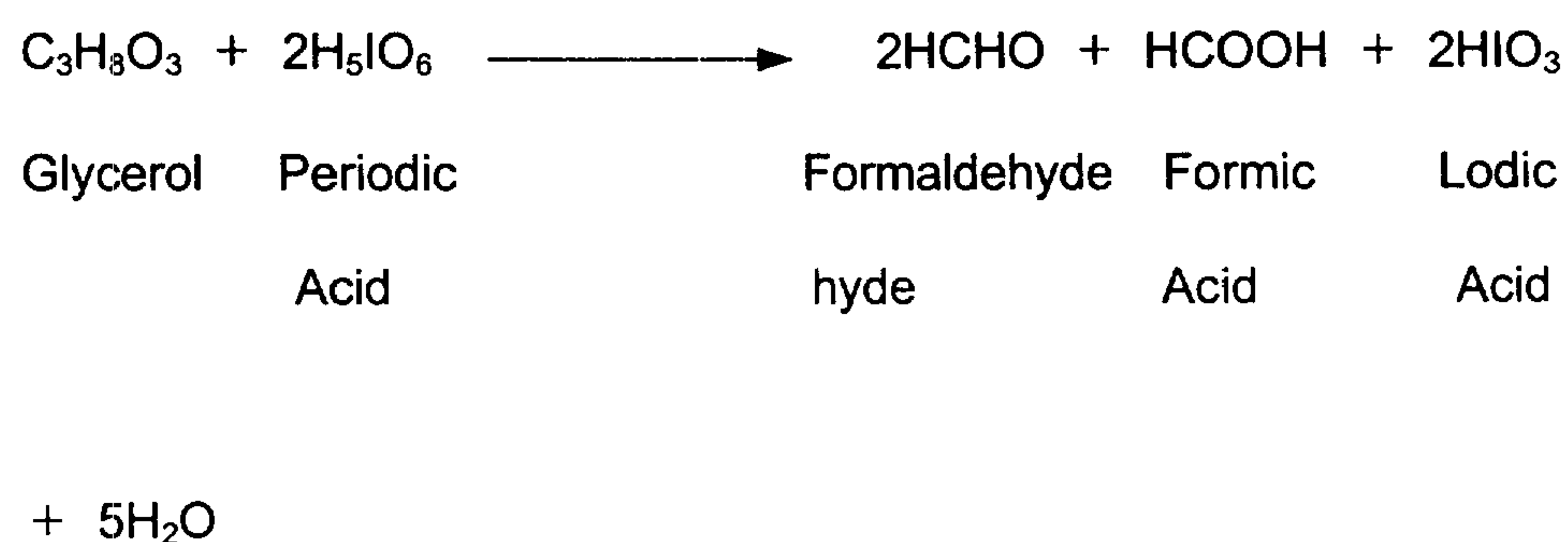
This standard shall specify the analysis method of Glycerol, Ethylene Glycol in alkyd resin solution.

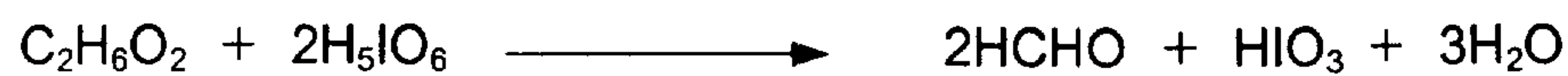
3-2 Method summary

Oxidized first hydroxy group of ethylene glycol and glycerol by periodic acid becomes formaldehyde.

Oxidized sec-hydroxyl group of glycerol becomes formic acid.

It may decide formic acid and formaldehyde with acid or iodine titration. You can calculate glycerol and ethylene glycol by algebraic equation in here.





Ethylene	Periodic	Formaldehyde	Iodic
Glycol	Acid	de	Acid

Penta erythritol is not react in here.

3-3 Equipment

- (1) Burette : 50 ml, 100 ml
- (2) Pipette : 20 ml, 50 ml
- (3) Beaker : 200 ml, 400 ml
- (4) Erlenmeyer flask : 1000 ml, the thing that has a lid
- (5) Volume flask : 100 ml
- (6) Mess cylinder : 10 ml
- (7) Filter paper
- (8) Watch glass

3-4 Reagent

- (1) Methyl purple indicator solution
- (2) Periodic acid (11 gr / ℓ) : After melting 11 gr of period acid(HIO₄) with water, and make 1 ℓ of solution. Store in a brown bottle at fresh air.
- (3) Iodine cally solution : Make 1 ℓ of water that is melted 200 gr of iodination cally
- (4) Sodium hydroxide standard solution (0.1 N)
- (5) Sodium thidsulfate standard solution (0.2 N)
- (6) Starch indicator solution: Make 1 ℓ of water that is melted 5gr of starch
- (7) Sulfuric acid (1 : 5)

3-5 Preparation

- (1) Analyze phthalic anhydride in alkyd resin and resin solution according to ASTM D 563.

Extract fatty acid according to clause 5 – 6 ~ 7 in ASTM D 1398, and does not throw away an aqueous solution.

(2) That glycerol and ethylene glycol is remained in an aqueous solution layer is based on the following order.

(a) Remove the solution into a 400 mL beaker, and evaporate it up to 60 mL

The beaker covers a watch glass while it is boiling.

(b) After cooling down with room temperature, filtrate into a 100 mL flask. Shake and stir it after filling up to indication line.

(c) Measure 20 mL, put into a 1 L flask, decant 2 drops of methyl purple indicator solution, and neutralize with sodium hydroxide. Add 50 mL of HIO_4 solution, stir a mixture after covering a lid.

(3) Prepare two official specimen that contain 20 mL of water, and leave them alone at room temperature for about 10 ~ 50 minutes.

(4) Pour 100 mL of water with the aqueous solution and official specimen of 3 - 5 (2) ②, decant 3 drops of methyl purple indicator, neutralize and titrate with 0.1 N sodium hydroxide.

(5) Pour 150 mL of water in titrated solution. After adding 30 mL of KI solution and 25 mL of sulfuric acid (1 : 5), titrate with 0.2 N $\text{Na}_2\text{S}_2\text{O}_3$.

You should use 10 mL of starch solution with indicator, and stop titration when blue color disappear.

3-6 Calculation

(1) Glycerol content : G

$$G = [(A - B) N \times 0.09206 / WF] \times 100$$

A : Sodium hydroxide titer of the specimen (mL)

B : Sodium hydroxide titer of official specimen (mL)