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Tall-oil fatty acids for paints and varnishes — Test methods and characteristic values

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee STI/3, Paints, media and related products.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Tall-oil fatty acids for paints and varnishes - Test methods and characteristic values (ISO 8623:2015)

Acides gras de tall-oil pour peintures et vernis - Méthodes d'essai et valeurs caractéristiques (ISO 8623:2015)

Tallöl-Fettsäuren für Beschichtungsstoffe - Prüfverfahren und Kennwerte (ISO 8623:2015)

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European foreword

This document (EN ISO 8623:2015) has been prepared by Technical Committee ISO/TC 35 “Paints and varnishes” in collaboration with Technical Committee CEN/TC 139 “Paints and varnishes” the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2016, and conflicting national standards shall be withdrawn at the latest by March 2016.

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The text of ISO 8623:2015 has been approved by CEN as EN ISO 8623:2015 without any modification.

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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The committee responsible for this document is ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 8623:1997), which has been technically revised with the following changes:

— the former requirements were changed to information on characteristic values;

— the data for unsaponifiable matter content was changed from mass fraction of max. 2,5 % to max. 5 %;

— the method for the determination of the unsaponifiable matter (diethyl ether method) was implemented from the 1980 edition of ISO 150 because it is no longer included in the actual revision of ISO 150, i.e. ISO 150:2006;

— the concentration of phenolphthalein indicator solution was changed from 10 g/l in ethanol to 5 g/l in ethanol or in a 1:1 ethanol/water mixture.

Introduction

Normally, requirements are agreed between the interested parties. So with this new edition, this International Standard no longer specifies requirements but only test methods and gives information on characteristic values for tall-oil fatty acids.

Tall-oil fatty acids for paints and varnishes — Test methods and characteristic values

1 Scope

This International Standard specifies test methods and gives information on characteristic values for distilled tall-oil fatty acids for paints and varnishes.

2 Normative references

The following documents, in whole or in part, are normally referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 383, *Laboratory glassware — Interchangeable conical ground joints*

ISO 2114, *Plastics (polyester resins) and paints and varnishes (binders) — Determination of partial acid value and total acid value*

ISO 2811 (all parts), *Paints and varnishes — Determination of density*

ISO 3681, *Binders for paints and varnishes — Determination of saponification value — Titrimetric method*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 3961, *Animal and vegetable fats and oils — Determination of iodine value*

ISO 4630, *Clear liquids — Estimation of colour by the Gardner colour scale*

3 Test methods and characteristic values

Test methods and typical characteristic values for tall-oil fatty acids for paints and varnishes are given in Table 1.

Table 1 — Test methods and characteristic values for tall-oil fatty acids

Property			Characteristic value	Test method
Density	at 20 °C	g/cm^3	0,900 to 0,910	ISO 2811
	at 23 °C	g/cm^3	0,898 to 0,908	
Colour			max. 5	ISO 4630
Acid value	mg KOH/g		min. 192	ISO 2114
Saponification value	mg KOH/g		min. 193	ISO 3681
Iodine value	g Iodine /100 g		min. 125	ISO 3961
Unsaponifiable matter	% (mass fraction)		max. 5	Annex A
Rosin acid content	% (mass fraction)		max. 2,5	Annex B

Annex A
(normative)

Determination of unsaponifiable matter (Diethyl ether method)

A.1 General

This test method is applicable to all fats. It is, however, only approximate for certain fats having high content of unsaponifiable matter.

A.2 Terms and definitions

A.2.1

unsaponifiable matter

substances soluble in the fat, which after saponification are insoluble in water but soluble in the solvent used for the determination

Note 1 to entry It includes lipids of natural origin such as sterols, alcohols and hydrocarbons as well as any foreign organic matter non-volatile at 100 °C (mineral oils) which may be present.

A.3 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following:

A.3.1 150 ml flask fitted with a reflux condenser.

A.3.2 500 ml separating funnels.

A.3.3 Oven, regulated at (103 ± 2) °C.

A.4 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 as defined in ISO 3696.

A.4.1 Aqueous solution of potassium hydroxide, c (KOH) = 28 g/l.

A.4.2 Ethanolic solution of potassium hydroxide, c (KOH) ≈ 112 g/l.

Dissolve 120 g potassium hydroxide in ethanol, c (C_2H_5OH) = 95 % volume fraction, and make up to 1 l. The reagent shall not be darker than straw yellow.

A.4.3 Diethyl ether, free from residue.

NOTE Diethyl ether is used as a solvent which generally gives higher result than light petroleum solvents.

A.5 Procedure

Weigh about 5 g of fat to within 0,01 g into the flask (A.3.1).

Add 50 ml of the ethanolic potassium hydroxide solution (A.4.2). Attach the condenser. Boil gently for an hour.

When heating is completed, wait for the solution to cool down and disconnect the condenser. Transfer the contents of the flask into a separating funnel (A.3.2), rinsing the flask with distilled water (100 ml in all). Transfer this water also in the separating funnel.

Rinse the flask and the condenser with 100 ml of diethyl ether and pour this into the separating funnel. Stopper and shake vigorously. While the contents are still slightly warm, hold vertically until there is a clean separation of the two layers. If an emulsion forms due to excess of alkalinity of the medium, add a few drops of hydrochloric acid, c (HCl) ≈ 37 g/l.

Draw off the aqueous ethanolic layer into the flask used for saponification.

Pour the ethereal extract through the neck of the funnel into another separating funnel containing 40 ml of water.

Using the first separation funnel, extract the aqueous ethanolic soap solution twice more, each time in the same way with 100 ml diethyl ether, and combine the ethereal fractions in the second funnel. If this ethereal solution contains suspended solid matter, filter carefully and wash the residue and the filter with a little diethyl ether to remove all soluble matter.

Rotate the funnel containing the combined extracts and the 40 ml of water without violent shaking and after the layers have separated draw off the wash water. Wash the ethereal solution twice with 40 ml water, shaking vigorously each time. Then, wash successively with 40 ml of the aqueous potassium hydroxide solution (A.4.1), 40 ml of water, and again with 40 ml of the aqueous potassium hydroxide solution (A.4.1), then at least twice more with 40 ml of water.

Continue to wash with water until the wash-water no longer gives a pink colour on the addition of a drop of phenolphthalein solution.

Pour off the ethereal solution quantitatively, a little at a time through the top of the separating funnel (washing the funnel with the solvent) into a 500 ml tared flask, and evaporate to small volume.

Add 6 ml of acetone, and remove the volatile solvent completely in a gentle current of air, holding the flask obliquely while turning it in a boiling water bath in which it is almost entirely submerged.

Complete the drying in a 103 °C oven (A.3.3) for 15 min, placing the flask in a horizontal position. Weigh after cooling in a desiccator.

Repeat the drying for successive 15 min periods until the loss of mass between two successive weighings is less than 0,1 %.

After weighing the residue, dissolve it in 20 ml of freshly distilled and neutralized ethanol [c (C_2H_5OH) = 95 % volume fraction]. Titrate with alcoholic KOH solution, c (KOH) = 0,1 mol/l, in the presence of phenolphthalein. If the volume used exceeds 0,2 ml, the determination shall be repeated.

A.6 Expression of results

Calculate the unsaponifiable matter, expressed as a mass fraction in percent, using the following formula:

$$\text{Unsaponifiable matter} = \frac{100\ m_1}{m_0} \tag{A.1}$$

where

m_0 is the mass of the test portion, in grams;

m_1 is the mass of the residue, in grams.

Annex B
(normative)

Determination of rosin acid content

B.1 Principle

A test portion is treated with butanol sulphuric acid esterification reagent under specified conditions and subsequently titrated against a standard volumetric potassium hydroxide solution, using phenolphthalein as indicator. The rosin acid content is calculated in terms of abietic acid.

B.2 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following:

B.2.1 Air condenser, 760 mm long, with a 24/29 ground glass cone.

B.2.2 Burette, automatic type, having a capacity of 25 ml, for the standard potassium hydroxide solution, fitted with soda lime traps to protect against absorption of atmospheric CO_2.

B.2.3 Conical flask, of borosilicate glass, of capacity 250 ml, with a 24/29 conical ground glass neck.

B.2.4 24/29 conical ground glass joints, complying with the requirements of ISO 383.

B.2.5 Moisture trap, of construction similar to that shown in Figure B.1 and wrapped with 12,5 mm heat-resistance tape.

Dimensions in millimetres

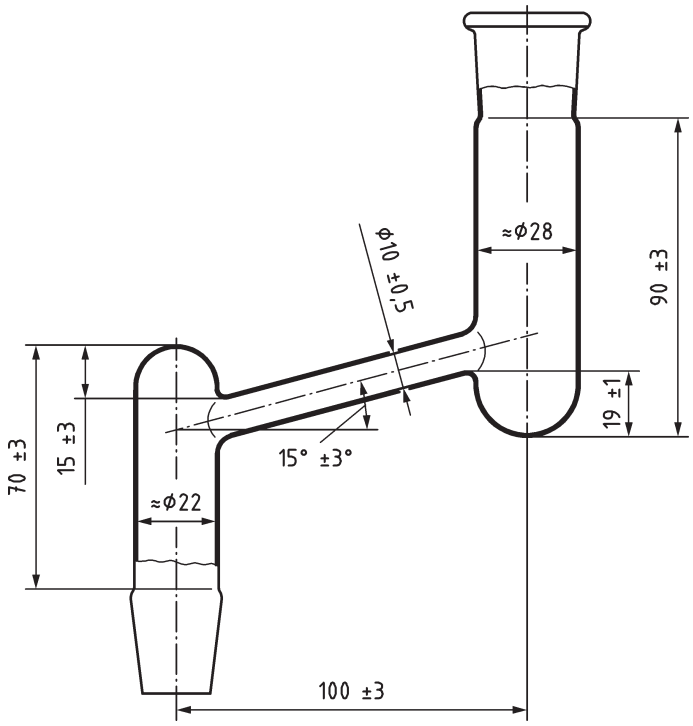

Figure B.1 — Moisture trap

B.2.6 Pipette, automatic, of capacity 50 ml.

B.3 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 as defined in ISO 3696.

B.3.1 Butanol sulphuric acid esterification reagent.

Introduce 500 ml of n-butanol, 500 ml of toluene, and 3,3 ml of concentrated sulphuric acid, ρ = 1,84 g/cm^3, into a 2 l round-bottom flask with ground-glass neck; connect to a moisture trap and condenser, and reflux in a heating mantle for 30 min to distil off the water. Cool and store in a glass-stoppered bottle.

B.3.2 Potassium hydroxide, standard volumetric methanolic solution, c (KOH) = 0,2 mol/l.

Dissolve 13,3 g of KOH pellets in 1 l of methanol. Standardize against a potassium hydrogen phthalate primary standard.

B.3.3 Phenolphthalein indicator solution, c = 5 g/l in ethanol, c (C_2H_5OH) = 95 % volume fraction, or in an ethanol/water mixture 1 + 1 (by volume).

B.4 Procedure

Carry out the determination in duplicate.

Weigh to the nearest 1 mg, 5 g to 8 g of the sample and transfer to a 250 ml conical flask (B.2.3). Using the automatic pipette (B.2.6), measure 50 ml of the esterification reagent (B.3.1) into the flask. Connect

the flask to the moisture trap (B.2.5) and condenser (B.2.1), place on a hotplate, heat to boiling, and reflux for 20 min.

At the end of the heating period, remove the flask from the hotplate and allow to cool to room temperature.

Add 0,5 ml of phenolphthalein solution (B.3.3) and titrate with the standard volumetric potassium hydroxide solution (B.3.2) until the solution turns to pink-red.

In the case of dark-coloured products or those containing small amounts of mineral acid or alkali, the end-point of the titration should preferably be determined potentiometrically.

Carry out a blank test, also using 50 ml of the esterification reagent.

B.5 Expression of results

Calculate the rosin acids content c_R, expressed as a mass fraction in percent, using the following formula:

$$c_R = \frac{(V_1 - V_2) \cdot c \times 302{,}4 \times 1{,}042}{m \times 10} - 0{,}1 \qquad \text{(B.1)}$$

where

V_1 is the volume, in millilitres, of the standard volumetric potassium hydroxide solution used for the determination;

V_2 is the volume, in millilitres, of the standard volumetric potassium hydroxide solution used for the blank test;

c is the concentration, in moles per litre, of the potassium hydroxide solution;

m is the mass, in grams, of the test portion;

302,4 is the molecular mass of abietic acid;

1,042 is an experimentally determined factor to correct for the slight esterification of rosin acids;

0,1 is an experimentally determined factor to correct for the unesterified acids.

If the two results (duplicates) differ by more than the value indicated in B.6, repeat the procedure.

Calculate the mean of two valid results (replicates) and report the result to one decimal place.

B.6 Repeatability

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method, can be expected to lie with a 95 % probability, is a mass fraction of 0,2 %.

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