INTERNATIONAL STANDARD

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Binders for paints and varnishes — Gum rosin — Gas-chromatographic analysis

Liants pour peintures et vernis — Colophane — Analyse par chromatographie en phase gazeuse

Reference number
ISO 19334:2010(E)

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 19334 was prepared by Technical Committee 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 19336:2006), Table 1 of which has been technically revised. The name of *Pinus keysia* was corrected and data for the rosin-acid composition of *Pinus yunnanensis* has been added.

Introduction

Gum rosins are important commercial materials, primarily composed of rosin acids, but also containing some neutral material. For many applications, it is necessary to know the level of the individual rosin acids present in these products. Gas chromatography has proven to be a useful tool for such determinations (see ASTM D 509), and capillary chromatography, specified in these test methods, is considered to be the most effective gas-chromatographic technique currently available.

Due to hydrogen bonding, unmodified rosin acids cannot be volatilized at atmospheric pressure without undergoing decomposition. So, it is necessary to convert the free acids to more volatile and more stable methyl esters, prior to chromatographic separation.

This International Standard describes one way to prepare methyl esters. The classic method is through the use of diazomethane, but diazomethane is a hazardous and toxic material, and so is no longer the preferred agent.

Trimethylphenylammonium hydroxide (TMPAH) and tetramethylammonium hydroxide (TMAH) are satisfactory methylating agents as both produce results that are very similar to those of diazomethane, but without the hazards that are associated with diazomethane.

N,*N*-dimethylformamide dimethyl acetal (DMF-DMA) gives results comparable to those obtained with TMPAH and TMAH and is easy and safe to use. However, the reagent is moisture-sensitive, requiring samples to be free of any significant levels of water.

This International Standard specifies the method using TMAH.

Binders for paints and varnishes — Gum rosin — Gas-chromatographic analysis

1 Scope

This International Standard specifies a gas-chromatographic method for determining the amounts of certain rosin acids in gum rosin using capillary gas-chromatographic separation of the volatile methyl esters of these acids. It is intended primarily to permit the identification of gum rosin from specific species of pine trees. It is not designed for the quantitative analysis of gum rosin. If such analyses are required, the internal-standard technique specified in ASTM D 5974 should be used.

2 Normative references

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

GB 8145, *Gum rosin — Test methods of rosin*

ASTM D 509, *Standard Test Methods of Sampling and Grading Rosin*

Naval Stores Act of 1923 (42 Stat 1435.7, USC 91-99) as amended in 1951

3 Sampling

Take a representative sample following the procedures specified in GB 8145, ASTM D 509 or the Naval Stores Act of 1923 (42 Stat 1435.7, USC 91-99) as amended in 1951.

NOTE Rosin samples need to be freshly broken from a larger mass to ensure the results are not affected by air oxidation of the rosin.

4 Apparatus

4.1 Gas chromatograph

Use an instrument equipped with a flame-ionization detector (FID) that can be operated under the conditions given in 7.1.

4.2 Column

The recommended (referee) column is a fused-silica BDS column 25 m in length, 0,32 mm in internal diameter and with a 0,20 µm film thickness, which gives separation equivalent to or better than that shown in Figures 1 to 3.

If other columns are used, the operating conditions shall be chosen such that the separation obtained is equivalent to or better than that shown in Figures 1 to 3.

5 Reagents and materials

Reagent-grade chemicals should preferably be used in all analyses. If other grades are used, it shall be first ascertained that the reagent concerned is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1 Tetramethylammonium hydroxide (TMAH) solution, 24 % in methanol, CAS No. 75-59-2

5.2 Tetramethylammonium hydroxide (TMAH) solution, 6 % in methanol. Dilute 25 ml of the reagent described in 5.1 with 75 ml of methanol.

5.3 Phenolphthalein solution, 1 % (mass per volume) in methanol

5.4 Methanol, anhydrous

5.5 Toluene (optional)

6 Preparation of methyl esters

Weigh approximately 50 mg of the sample into a suitable vial, and dissolve it in 0,5 ml to 3,0 ml of methanol. Add 2 to 3 drops of phenolphthalein indicator solution, and titrate to a pink colour (pH 7,8 to 8,1) with the solution of 6 % solution. When the solution is injected into the heated injection port of the chromatograph, the tetramethylammonium salts are pyrolyzed to methyl esters.

NOTE For solid rosin, 2 to 3 drops of toluene may be added to the vial prior to the addition of TMAH, to assist in dissolving the sample.

7 Gas-chromatographic analysis

7.1 Set-up of gas chromatograph (GC)

Set the GC conditions so that they are approximately as follows (see also the last paragraph in this subclause):

Column temperature:	195 °C, isothermal
Injection port temperature:	300 °C
Injection port liner:	glass splitter
Detector temperature:	280 °C
Carrier gas:	hydrogen or helium
Linear flow rate:	40 cm/s
Split ratio:	40 to 1
Detector:	FID
	hydrogen: 50 kPa
	air: 100 kPa

These are the typical operating conditions for the instrument using the BDS column. The particular equipment used shall be adjusted in accordance with the manufacturer's instructions to optimize its performance. Ongoing adjustment of operating temperature and flow rate may be necessary to maintain optimum performance of the column due to ageing.

7.2 Analysis

Inject 0,5 µl to 1,0 µl of the test sample prepared in Clause 6.

NOTE Dilution of the sample with additional solvent may be necessary to obtain injections that do not overload the column or detector.

7.3 Calculation

Sum all the areas of the individual peaks, exclusive of the solvent peak, to obtain the total peak area.

Calculate the relative percentage of each rosin acid methyl ester present, uncorrected for the amount of polymeric materials present, using the equation

$$\text{Rosin acid}(\%) = \frac{A \times 100}{A_{\text{Tot}}}$$

where

A is the peak area for the rosin acid methyl ester being determined;

A_{Tot} is the sum of the areas of all rosin acid methyl ester peaks.

NOTE See Figures 1 to 3 for typical chromatograms of selected gum rosins and Table 1 for their compositions determined from these and other such chromatograms.

8 Test report

The test report shall contain at least the following information:

a) all details to identify the product tested;

b) reference to this International Standard (ISO 19334:2010);

c) the area percent of individual rosin acids to the nearest 0,1 %;

d) by comparison with Table 1 and Figures 1 to 3, the identity of the gum rosin;

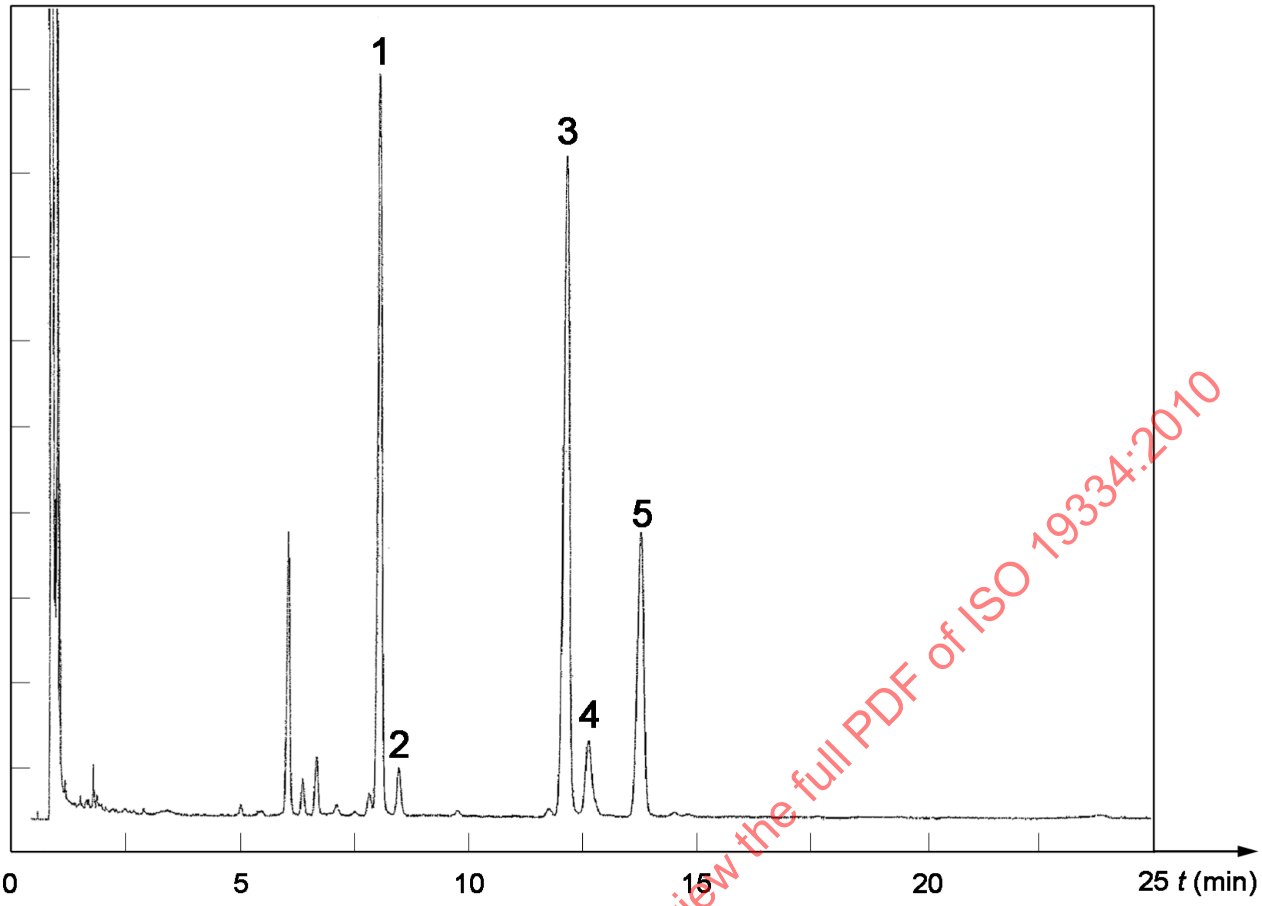
e) any deviation from the test method specified;

f) the date of the test.

Table 1 — Typical contents of characteristic rosin acids in gum rosins from pine species
(determined by the gas-chromatographic method specified in this International Standard)

Rosin acid	***Pinus massoniana*** %	***Pinus merkusii*** %	***Pinus elliottii*** %	***Pinus pinaster*** %	***Pinus caribaea*** %	***Pinus keysia*** %	***Pinus yunnanensis*** %	**Relative Retention**
Communic	0	0	5	0	1	0	0	0,588
Palustric	25	20	19	19	22	20	26	0,634
Isopimaric	2	15	17	5	8	4	6	0,664
Abietic	35	35	23	38	35	41	31	0,967
Dehydroabietic	4	5	5	6	7	4	5	1,000
Neoabietic	16	5	20	16	17	18	22	1,092
Mercusic	0	7	0	0	0	0	0	1,514

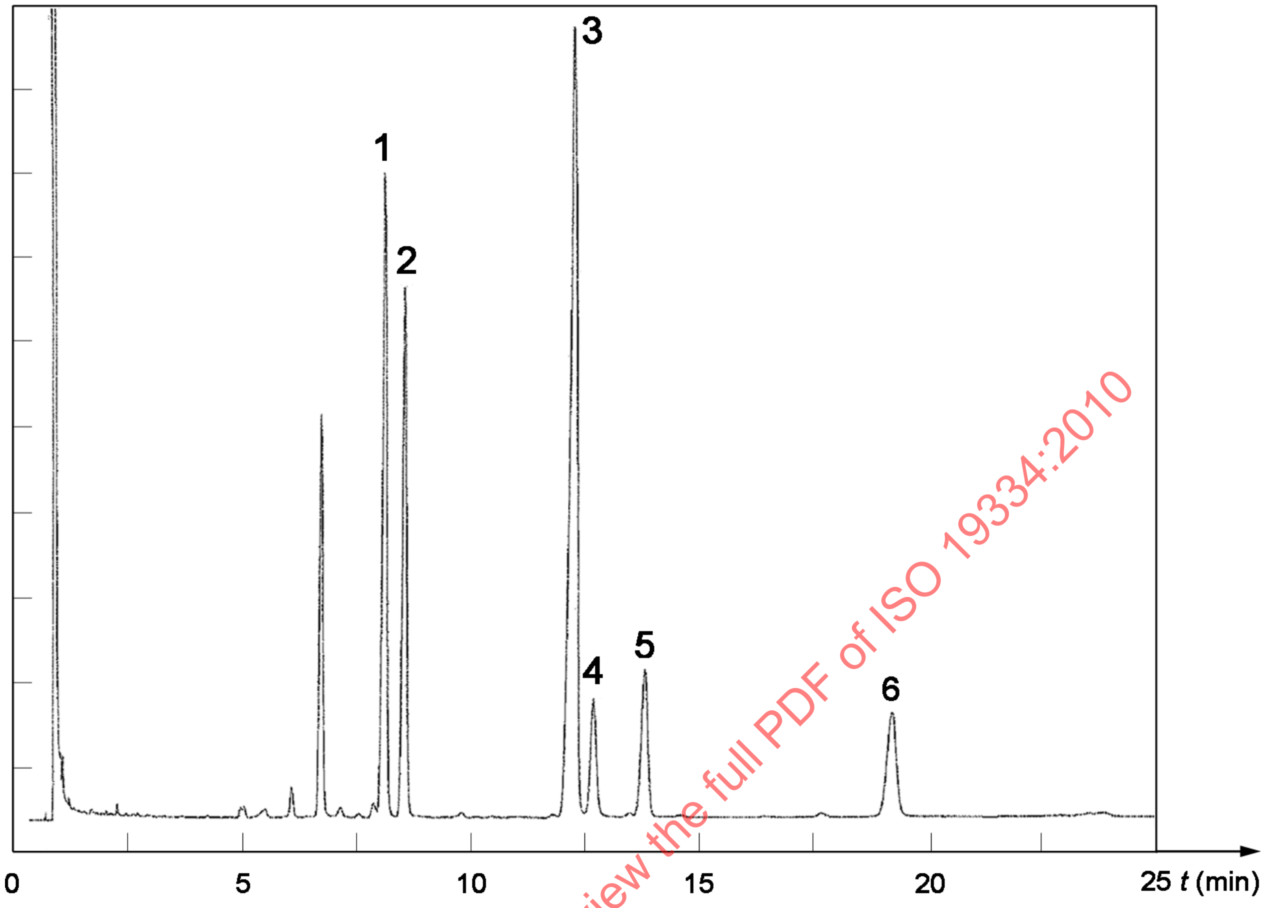
NOTE Capillary gas-liquid chromatography (GLC). Labelled peaks eluated as methyl esters. Conversion of rosin acids to their methyl deviates by means of tetramethylammonium hydroxide (TMAH) procedure. Column: fused silica BDS, $l = 25$ m, $d = 0{,}32$ mm, film thickness = 0,2 µm, $T = 195$ °C (isothermal), carrier gas H_2 at 50 kPa.



Key

1	palustric acid
2	isopimaric acid
3	abietic acid
4	dehydroabietic acid
5	neoabietic acid

Figure 1 — Chromatogram of rosin acid methyl esters from *Pinus massoniana* rosin



Key

1	palustric acid
2	isopimaric acid
3	abietic acid
4	dehydroabietic acid
5	neoabietic acid
6	mercusic acid

Figure 2 — Chromatogram of resin acid methyl esters from *Pinus merkusii* rosin