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

*Liants pour peintures et vernis — Gomme-résine — Analyse par  
chromatographie en phase gazeuse*



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## Foreword

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ISO 19334 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

## 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, *Standard Test Methods of Sampling and Grading Rosin*), 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 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 a 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 Preparation of methyl esters

### 4.1 Apparatus

#### 4.1.1 Gas chromatograph

Use an instrument equipped with a flame-ionization detector (FID) that can be operated under the conditions given in 5.1.

#### 4.1.2 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  $\mu\text{m}$  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.