BS ISO 4629-3:2018



BSI Standards Publication

Binders for paints and varnishes — Determination of hydroxyl value

Part 3: Rapid test



National foreword

This British Standard is the UK implementation of ISO 4629-3:2018.

The UK participation in its preparation was entrusted to Technical Committee STI/10, Test methods for paints.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Part 3: Rapid test

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Partie 3: Méthode rapide



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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.

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see <u>www.iso.</u> <u>org/iso/foreword.html</u>.

This document was prepared by Technical Committee ISO/TC 35, Paints and varnishes.

A list of all parts in the ISO 4629 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at <u>www.iso.org/members.html</u>.

Introduction

The most frequently described method for determining the hydroxyl number is conversion with acetic anhydride in pyridine with subsequent titration of the released acetic acid.

 $\mathrm{H_{3}C\text{-}CO\text{-}O\text{-}CH_{3}+R\text{-}OH} \ \rightarrow \ \mathrm{R\text{-}O\text{-}CO\text{-}CH_{3}+CH_{3}COOH}$

However, this method suffers from the following disadvantages:

- the sample is boiled under reflux for 1 h;
- the method cannot be automated;
- small hydroxyl numbers cannot be determined exactly;
- unpleasant and toxic pyridine is used.

For these reasons, a far simpler method was selected for automation. The (primary and secondary) hydroxyl groups are converted to acidic carbamate groups using toluene-4-sulfonyl-isocyanate.

 $H_3C-(C_6H_4)-SO_2-NCO + R-OH \rightarrow H_3C-(C_6H_4)-SO_2-NH-COOR$

The carbamate can then be titrated with the strong base tetrabutylammonium hydroxide (TBAOH) under non-aqueous conditions, i.e. in an organic solvent.

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1 Scope

This document specifies a titrimetric method for determining the hydroxyl groups in resins and binders for paints and varnishes.

This method is primarily suitable for neutral media. Acidic products provide higher values; neutral products provide, through neutralization of the acidic carbamates, lower values. For these products, preliminary tests are performed to ensure the applicability of the method.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 4618, Paints and varnishes — Terms and definitions

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

ISO 15528, Paints, varnishes and raw materials for paints and varnishes — Sampling

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at <u>http://www.electropedia.org/</u>

3.1

hydroxyl value

number of milligrams of potassium hydroxide (KOH) corresponding to hydroxyl groups that have been acetylated under specified test conditions in 1 g of the product tested

[SOURCE: ISO 4629-1:2016, 3.1]

4 Principle

The primary and secondary hydroxyl groups of the sample are converted to acidic carbamate groups using toluene-4-sulfonyl-isocyanate. The carbamate can then be titrated with the strong base tetrabutylammonium hydroxide (TBAOH) under non-aqueous conditions, i.e. in an organic solvent.