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# International Standard



# 4326

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

## Non-ionic surface active agents — Polyethoxylated derivatives — Determination of hydroxyl value — Acetic anhydride method

*Agents de surface non ioniques — Dérivés polyéthoxylés — Détermination de l'indice d'hydroxyle — Méthode à l'anhydride acétique*

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## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4326 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in June 1976.

It has been approved by the member bodies of the following countries :

Australia	Ireland	Romania
Austria	Japan	South Africa, Rep. of
Belgium	Korea, Rep. of	Switzerland
Brazil	Mexico	Turkey
Chile	Netherlands	United Kingdom
France	New Zealand	USA
Germany, F.R.	Philippines	USSR
India	Poland	

The member body of the following country expressed disapproval of the document on technical grounds :

Spain

# Non-ionic surface active agents — Polyethoxylated derivatives — Determination of hydroxyl value — Acetic anhydride method

## 0 INTRODUCTION

The classical method for the determination of the hydroxyl value described in this International Standard requires, because of the toxic effects of pyridine and acetic anhydride, that all handling and the titration be conducted under a well ventilated fume hood. However, for information, it is pointed out that annex C of ISO 4327 provides a procedure using a special apparatus which reduces the danger due to pyridine and acetic anhydride.

## 1 SCOPE

This International Standard specifies a method for the determination of the hydroxyl value of polyethoxylated condensates by esterification of the hydroxyl groups with acetic anhydride.

## 2 FIELD OF APPLICATION

This method is applicable to the determination of the hydroxyl groups of polyethoxylated condensates of aliphatic and alicyclic compounds (in particular, of ethylene oxide adducts of primary fatty alcohols, of alkylphenols and of fatty acids) and can be used for the determination of hydroxyl values from 10 to 1 000.

However, certain substances present in these materials may react with the acetic anhydride or with the standard volumetric sodium hydroxide solution used and consequently falsify the results.

The possible interferences are detailed below :

- Primary and secondary amines, amides, tertiary alcohols, thiols and epoxides undergo side reactions which affect the accuracy of the method.
- Long-chain fatty acids and esters may interfere by forming anhydrides which are more stable than acetic anhydride and are not completely decomposed at the end of the method.
- Other free acids interfere by reacting with the standard volumetric sodium hydroxide solution; bases,

including some tertiary amines, interfere by reacting with the acetic acid produced. In these cases a correction may be made for the acidity or alkalinity (see ISO 4314).

Epoxides are known to interfere, but the method can still be used if it is possible to eliminate them, by cold vacuum distillation, without affecting the hydroxyl value. By this means, free ethylene oxide, which interferes at levels above 0,5 % (*m/m*), can be eliminated.

The presence of water in the sample is revealed by reaction with the acetic anhydride, but the method may nevertheless be used without risk if the precautions described in the procedure are followed.

This method is not applicable to propoxylated products.

## 3 REFERENCES

ISO 607, *Surface active agents and detergents — Methods of sample division.*

ISO 2211, *Liquid chemical products — Measurement of colour in Hazen units (platinum-cobalt scale).*

ISO 4314, *Surface active agents — Determination of free alkalinity or free acidity — Titrimetric method.*

ISO 4317, *Surface active agents — Determination of water content — Karl Fischer method.*

ISO 4327, *Non-ionic surface active agents — Polyalkoxylated derivatives — Determination of hydroxyl value — Phthalic anhydride method.*

## 4 DEFINITION

**hydroxyl value  $I(\text{OH})$**  : The number of milligrams of potassium hydroxide needed to neutralize the acetic acid required to acetylate the hydroxyl groups in 1 g of the material, or the number of milligrams of potassium hydroxide corresponding to the hydroxyl groups in 1 g of the material.

NOTE — The number of moles of potassium hydroxide corresponding to the hydroxyl value is equal to the number of hydroxyl groups present in 1 kg of the material.