# SLOVENSKI STANDARD
# SIST-TP CEN/TR 15314:2006

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**Trajnost lesa in lesnih proizvodov – Kvantitativno določevanje kvarternih amonijevih spojin v lesu**

Durability of wood and wood-based products - Quantitative determination of quaternary ammonium compounds in wood

Dauerhaftigkeit von Holz und Holzprodukten - Quantitative Bestimmung von quartären Ammoniumverbindungen in Holz



Durabilité du bois et des matériaux dérivés du bois - Détermination quantitative des composés ammonium quaternaire dans le bois



**Ta slovenski standard je istoveten z:** **CEN/TR 15314:2006**

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**ICS:**

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| 79.040 | Les, hlodovina in žagan les | Wood, sawlogs and sawn timber |

**SIST-TP CEN/TR 15314:2006** **en**

TECHNICAL REPORT

RAPPORT TECHNIQUE

TECHNISCHER BERICHT

# CEN/TR 15314

January 2006

ICS 79.080

English Version

# Durability of wood and wood-based products - Quantitative determination of quaternary ammonium compounds in wood

Durabilité du bois et des matériaux dérivés du bois - Détermination quantitative des composés ammonium quaternaire dans le bois

Dauerhaftigkeit von Holz und Holzprodukten - Quantitative Bestimmung von quartären Ammoniumverbindungen in Holz

This Technical Report was approved by CEN on 14 December 2005. It has been drawn up by the Technical Committee CEN/TC 38.

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## Foreword

This Technical Report (CEN/TR 15314:2006) has been prepared by Technical Committee CEN/TC 38 “Durability of wood and wood-based products”, the secretariat of which is held by AFNOR.

## Introduction

At present, no standardised method for the analysis of quaternary ammonium compounds (QAC) in wood is recognised in Europe. Only a few national standards are available world wide, e.g. ASTM D5584-94, AWPA A 16-93 or AWPA A 18-04.

This CEN Technical Report has been issued in order to facilitate the analysis of QAC-treated wood.

## 1 Scope

This CEN Technical Report specifies a laboratory method of determining the content of quaternary ammonium compounds in commercially QAC-treated wood. The method described has a measurement range up to QAC contents of 1 500 mg/kg of dry matter.

NOTE 1 This method may need some modifications with some wood species such as hardwoods.

NOTE 2 It is applicable to QAC with a molar mass ranging between 200 g/mol and 500 g/mol.

NOTE 3 The method has a quantification limit corresponding to 250 mg of QAC per kilogram of wood expressed as dry matter.

## 2 Normative references

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

EN 322, *Wood-based panel — Determination of moisture content*

EN ISO 1042, *Laboratory glassware — One-mark volumetric flasks (ISO 1042:1998)*



EN ISO 2871-2:1994, *Surface active agents — Detergents — Determination of cationic-active matter content — Part 2: Cationic active matter of low molecular mass (between 200 and 500) (ISO 2871-2:1990)*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*



EN ISO 4788, *Laboratory glassware —Graduated measuring cylinders (ISO 4788:2005)*

ISO 385-1, *Laboratory glassware — Burettes — Part 1: General requirements*

ISO 648, *Laboratory glassware —One-mark pipettes*

ISO 835-2, *Laboratory glassware — Graduated pipettes — Part 2: Pipettes for which no waiting time is specified*

## 3 Safety precautions

Persons using this method shall be familiar with normal analytical laboratory procedures and practice.

This method does not purport to address all the safety problems, if any, associated with its use.

It is the responsibility of the user to establish health and safety practices and to ensure compliance with any.

European or national regulatory conditions (also see Annex B for environmental, health and safety precautions) shall be taken into account.

## 4 Principle

The quaternary ammonium compound is extracted from the wood material using a mixture of methanol and hydrochloric acid in an ultrasonic bath.

The quaternary ammonium compound (cationic substance) is quantified by titration with an anionic surface active agent standard solution (sodium dodecyl sulfate) containing a mixture of a cationic and an anionic dye. The titration is performed in a 2-phase system consisting of water and trichloromethane according to EN ISO 2871-2. Cationic substances and the anionic dye (disulphine blue VN 150) constitute a salt which is soluble in trichloromethane and results in a blue colour.

The anionic dye in the salt is replaced gradually by the anionic surface active agent during the titration process leading to a discoloration of the organic phase at equivalence point. At the same time the disulphine blue moves into the water.

NOTE 1 Cationic surface active agents as well as other disulphine blue active substances will also react with the titrant and be determined as QAC.

NOTE 2 In order to determine the mass of QAC its molar mass is required.

## 5 Reagents

During the analysis, unless, otherwise specified, use only reagents of recognised analytical grade that have been checked in advance as to not interfere with the analytical results, and water complying with grade 3 as defined in EN ISO 3696.

**5.1 Trichloromethane** (CAS 67-66-3) ($CHCl_3$).

**5.2 Methanol** ($CH_3OH$).



NOTE Other solvents may be used instead of methanol as some extraction difficulties can occur with some wood species (e.g. hardwoods). It is recommended to cross check the extraction efficiency of any other solvent or solvent mixture with that of methanol / hydrochloric acid used.



**5.3 Dimidiumbromide** (CAS 518-67-2) ($C_{20}H_{18}BrN_3$).

**5.4 Disulphine blue VN 150** (CAS 129-17-9) ($C_{27}H_{31}N_2S_2O_6Na$).

**5.5 Sodium dodecyl sulfate** for analysis of surface active agents (CAS 151-21-3) ($C_{12}H_{25}SO_4Na$).

**5.6 Hydrochloric acid solution,** $c$(HCl) = 1 mol/l.

Dilute 9 ml of concentrated hydrochloric acid ($\rho_{20}$ = 1,18 g/ml) to 100 ml with water.

NOTE Test ampoules containing a definite amount may be used for the preparation of the hydrochloric acid solution instead of concentrated hydrochloric acid.

**5.7 Ethanol,** ($C_2H_5OH$) aqueous solution volume fraction 10 %.

Add 30 ml of ethanol to 270 ml of water and mix well.

**5.8 Sulfuric acid,** solution $c$($H_2SO_4$)= 2,5 mol/l.

Cautiously add, with stirring and cooling, 14 ml of concentrated sulfuric acid ($\rho_{20}$ = 1,84 g/ml) to about 80 ml of water and dilute to 100 ml with water.

NOTE Test ampoules containing a definite amount may be used for the preparation of the sulfuric acid solution instead of concentrated sulfuric acid.

## 6 Apparatus

Ordinary laboratory apparatus and the following.

NOTE Glassware should be thoroughly cleaned prior to use by means of ethanol.

**6.1 Analytical balance,** accurate to 0,1 mg.

**6.2 Ultra-sonic bath.**

**6.3 Volumetric glassware**, of class A quality in accordance with ISO 385-1 for the burettes, ISO 835-2 and ISO 648 for the pipettes, EN ISO 4788 for the measuring cylinders and EN ISO 1042 for the volumetric flasks. The burette shall be 10 ml graduated in 0,02 ml.

**6.4 Conical flasks with glass stopper,** 100 ml capacity.

**6.5 Polytetrafluoroethylene (PTFE) filter,** porosity 0,45 µm (e.g. combined with a syringe).

**6.6 Variable dispenser** (5ml to 30ml).

## 7 Preparation of the test sample



Collect at least 20 g of the sample material taken according to e.g. EN 212. This sample material is preferably ground under mild conditions to a powder with a particle size of less than 0,5 mm diameter.

Homogenise the ground material to obtain a representative sample, and store it in a brown glass bottle with screw caps with a polytetrafluoroethylene (PTFE) insert. This is the test sample.



## 8 Procedure

### 8.1 General

It is recommended to carry out at least two parallel analyses. If the results differ by more than 10 % an additional analysis shall be made.

### 8.2 Standard solutions

#### 8.2.1 Sodium dodecyl sulfate solution

Dissolve 1,442 g sodium dodecyl sulfate (5.5) in water into a 1 000 ml volumetric flask (6.3) resulting in a concentration of 0,005 mol/l.

NOTE The purity of the sodium dodecyl sulfate may be determined by the method given in 4.2.1 of EN ISO 2871-2:1994.

#### 8.2.2 Indicator solution

Weigh (500 ± 5) mg dimidiumbromide (5.3) into a 100 ml beaker and dissolve it in 30 ml hot aqueous ethanol (5.7). Then weigh (250 ± 5) mg disulphine blue VN 150 (5.4) into a 100 ml beaker and dissolve it in 30 ml hot aqueous ethanol (5.7). After cooling, transfer both solutions quantitatively into a 250 ml volumetric flask and make up to the mark with aqueous ethanol. Transfer 20 ml of this solution into a 500 ml volumetric flask which already contains approximately 200 ml water. Finally add 20 ml of 2,5 mol/l sulfuric acid (5.8) and make up to the mark with water.