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CSA W213:22
National Standard of Canada

Lignin — Quantification of hydroxyl groups by ^{31}P NMR

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CSA W213:22
January 2022

Title: *Lignin — Quantification of hydroxyl groups by ^{31}P NMR*

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National Standard of Canada

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Published in January 2022 by CSA Group
A not-for-profit private sector organization
178 Rexdale Boulevard, Toronto, Ontario, Canada M9W 1R3

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ICS 79.020
ISBN 978-1-4883-4027-7

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Preface

This is the first edition of CSA W213, *Lignin — Quantification of hydroxyl groups by ^{31}P NMR*.

CSA Group acknowledges that the development of this Standard was made possible, in part, by the financial support of the Standards Council of Canada, as part of the Innovation Initiative.

This Standard was prepared by the Technical Committee on Standards for Lignin, under the jurisdiction of the Strategic Steering Committee on Natural Resources, and has been formally approved by the Technical Committee.

This Standard has been developed in compliance with Standards Council of Canada requirements for National Standards of Canada. It has been published as a National Standard of Canada by CSA Group.

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0 Introduction

Lignin exists naturally in plants and trees and is one of the main components in wood. Given its abundance and its aromatic structure, lignin has the potential to replace fossil-based starting materials in a range of products including polymeric materials and fine chemicals. It is currently being evaluated by companies in Canada and around the world as an alternative to petroleum-based chemicals for products such as carbon fibres, flavourings, nutraceutical and pharmaceutical ingredients, resins, foams, rubber additives, and thermoplastics.

Lignin is a highly branched heterogeneous polyphenolic biopolymer composed of aromatic moieties with propyl side chains linked together by C-C and ether linkages. It contains various functional groups, including carbonyl groups, quinonoid groups, and aliphatic, phenolic, and carboxylic hydroxyl groups, the content of which is critical in determining lignin application, whether as a filler in polymer blends or as a raw material in polymer synthesis.

The majority of world commerce is governed by regulations-based product Standards. An absence of Standards for products and properties therefore limits market access. With international interest and on-going work in developing and commercializing new products derived from lignin, a strong knowledge of the physicochemical properties of lignin, including its chemical structure, functional groups, molecular weight (MW) distribution, and thermal properties, is required.

CSA Group has recently published two standard methods for the measurement of the glass transition temperature (T_g) and the thermal stability of kraft lignins (CSA W206 and CSA W207), which are two parameters of special relevance in the drying and processing of lignin for a range of chemical and polymer industry processes.

The method described in this Standard aims to provide Canadian producers with a standard method to quantify the hydroxyl groups present in lignins since this is of importance in several applications. It will provide lignin producers with an advantageous means for access to the domestic and global marketplaces.

1 Scope

1.1 General

This Standard describes a method for the quantification of the aliphatic (R–OH), carboxylic acid (R–COOH), and phenolic (Ph–OH) hydroxyl groups present in lignin. Phenolic hydroxyl groups include
a) syringyl (S), guaiacyl (G), and *p*-hydroxyphenyl (H) units; and
b) condensed phenolics incorporating β-5, 4-O-5', and 5-5' inter-unit linkages.

