Irish Standard
I.S. EN 16466-1:2013

Vinegar - Isotopic analysis of acetic acid and water - Part 1: ^{2}H-NMR analysis of acetic acid

I.S. EN 16466-1:2013

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Vinegar - Isotopic analysis of acetic acid and water - Part 1: ^{2}H-NMR analysis of acetic acid

Vinaigre - Analyse isotopique de l'acide acétique et de l'eau - Partie 1: Analyse RMN-^{2}H de l'acide acétique

Essig - Isotopenanalyse von Essigsäure und Wasser - Teil 1: ^{2}H-NMR-Analyse von Essigsäure

This European Standard was approved by CEN on 3 November 2012.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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Foreword

This document (EN 16466-1:2013) has been based on an international collaborative study of the methods published in Analytica Chimica Acta 649 (2009) 98-105, and organised under the auspices of the Permanent International Vinegar Committee (CPIV, Brussels).

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2013, and conflicting national standards shall be withdrawn at the latest by July 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

The European standard, *Vinegar — Isotopic analysis of acetic acid and water*, consists of the following parts:

— *Part 1: ^{2}H-NMR analysis of acetic acid*;

— *Part 2: ^{13}C-IRMS analysis of acetic acid*;

— *Part 3: ^{18}O-IRMS analysis of water*.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Vinegar is defined in EN 13188 as the acetic acid solution resulting from a double fermentation:

a) transformation of sugars to ethanol and

b) transformation of ethanol to acetic acid.

Conversely, EN 13189 defines acetic acid as "Product made from materials of non-agricultural origin".

Wine vinegar is defined by the European Regulations 479/2008 and 491/2009 as the product obtained exclusively from the acetous fermentation of wine, which is in turn defined as the product exclusively obtained from the alcoholic fermentation of fresh grapes, whether crushed or not, or of grape must.

In all types of vinegar, both the ethanol and the acetic acid should be obtained by a biotechnological process, and the use of acetic acids obtained from either petroleum derivatives or the pyrolysis of wood is not permitted according to the above definitions.

The isotopic analysis of acetic acid extracted from vinegar by ^{2}H-SNIF-NMR and ^{13}C-IRMS enables the distinction of grape origin from other sources, such as beet, cane, malt, apple and synthesis [1].

1 Scope

This European Standard specifies an isotopic method to control the authenticity of vinegar. This method is applicable on acetic acid of vinegar (from wine, cider, agricultural alcohol, etc.) in order to characterise the botanical origin of acetic acid and to detect adulterations of vinegar using synthetic acetic acid or acetic acid from a non-allowed origin (together with the method described in EN 16466-2).

The isotopic analysis of the extracted acetic acid by ^{2}H-NMR is based on a similar method already normalised for wine analysis [2].

This European Standard is not applicable to complex matrices made with vinegar as an ingredient, such as balsamic vinegar.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

Not applicable

3 Principle

The acetic acid from vinegar is first extracted with diethyl ether (or alternatively another solvent with similar properties such as tert-butyl methyl ether), using a liquid-liquid extractor, during at least 5 h. The solvent is then eliminated by distillation. The water content of the residue can be determined by the Karl Fischer method, or alternatively the acetic acid content may be determined by ^{1}H-NMR [3, 4]. The presence of organic impurities in the residue shall be checked e.g. on the basis of ^{1}H-NMR analysis or by GC analysis. The isotopic ratio of hydrogen atoms at the methyl site of acetic acid, $(D/H)_{CH3}$, is then determined by Nuclear Magnetic Resonance analysis of the Deuterium in the acetic acid extracted from the vinegar.

In case a correction is applied to the $(D/H)_{CH3}$ result to correct for organic impurities, this should be stated in the analytical report.

4 Reagents

All reagents and consumables used shall meet stated requirements of the used method/apparatus (as specified by the manufacturer). However, all reagents and consumables can be replaced by items with similar performance.

4.1 Diethyl ether

For analysis.

4.2 Standard N,N-tetramethylurea (TMU)

Standard TMU with a calibrated isotope ratio D/H.

4.3 Hexafluorobenzene (C_6F_6)

Used as field-frequency stabilisation substance (lock).