



**NSAI**  
Standards

Irish Standard  
I.S. EN 16466-2:2013

# Vinegar - Isotopic analysis of acetic acid and water - Part 2: $^{13}\text{C}$ -IRMS analysis of acetic acid

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## I.S. EN 16466-2:2013

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**NSAI**  
1 Swift Square,  
Northwood, Santry  
Dublin 9

T +353 1 807 3800  
F +353 1 807 3838  
E standards@nsai.ie  
W NSAI.ie

**Sales:**  
T +353 1 857 6730  
F +353 1 857 6729  
W standards.ie

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Vinegar - Isotopic analysis of acetic acid and water - Part 2:  
<sup>13</sup>C-IRMS analysis of acetic acid

Vinaigre - Analyse isotopique de l'acide acétique et de l'eau -  
Partie 2 : Analyse SMRI-<sup>13</sup>C de l'acide acétique

Essig - Isotopenanalyse von Essigsäure und Wasser - Teil 2:  
<sup>13</sup>C-IRMS-Analyse von Essigsäure

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Management Centre: Avenue Marnix 17, B-1000 Brussels

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## Foreword

This document (EN 16466-2:2013) 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of an identical text or by endorsement, at the latest by July 2013, and conflicting national standards shall be withdrawn at the latest by July 2013.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:  $^2\text{H}$ -NMR analysis of acetic acid;*
- *Part 2:  $^{13}\text{C}$ -IRMS analysis of acetic acid;*
- *Part 3:  $^{18}\text{O}$ -IRMS analysis of water.*

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## **Introduction**

Vinegar is defined by 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  $^2\text{H}$ -SNIF-NMR and  $^{13}\text{C}$ -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 cider, alcohol, wine, 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 not allowed origin (together with the method described in EN 16466-1).

The isotopic analysis of the extracted acetic acid by  $^{13}\text{C}$ -IRMS is based on a similar method already normalised for wine analysis [2].

## 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  $^{13}\text{C}/^{12}\text{C}$  ratio of acetic acid from vinegar is then determined by Isotope Ratio Mass Spectrometry (IRMS) on the  $\text{CO}_2$  gas resulting from a complete combustion at high temperature in an Elemental Analyser.

## 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 Carbon dioxide

For analysis, used as secondary reference gas for the determination of  $^{13}\text{C}/^{12}\text{C}$  ratio. Purity 5.2 minimum.

### 4.3 Helium

For analysis. Purity 5.6 minimum.

### 4.4 Oxygen

For analysis. Purity 5.0 minimum.

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