

**Vinegar - Isotopic analysis of acetic acid and water -
Part 3: 18O-IRMS analysis of water in wine vinegar**

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NATIONAL FOREWORD

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English Version

Vinegar - Isotopic analysis of acetic acid and water - Part 3: ¹⁸O-IRMS analysis of water in wine vinegar

Vinaigre - Analyse isotopique de l'acide acétique et de l'eau -
Partie 3: Analyse SMRI-¹⁸O de l'eau dans le vinaigre de vin

Essig - Isotopenanalyse von Essig und Wasser - Teil 3:
¹⁸O-IRMS-Analyse von Wasser in Weinessig

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

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Foreword

This document (EN 16466-3:2013) has been based on an international collaborative study of the method 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of an identical text or by endorsement, at the latest by July 2013, and conflicting national standards shall be withdrawn at the latest by July 2013.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: ^2H -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

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 accordance with this, it is clear that the production of wine vinegar by fermenting dried grapes and rehydrating with tap water is not allowed by European Regulations.

The isotopic analysis of water from vinegar by ^{18}O -IRMS enables the distinction of wine vinegar and vinegars from fermented dried grapes which have been rehydrated with water [1], and more generally helps to check the authenticity of wine vinegar.

1 Scope

This European Standard specifies an isotopic method to control the authenticity of wine vinegar. This method is applicable on wine vinegar in order to characterise the $^{18}\text{O}/^{16}\text{O}$ ratio of water, and allows differentiating wine vinegar from vinegars made from raisins or alcohol vinegar.

NOTE The Oxygen 18 isotopic analysis of water from vinegar 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 $^{18}\text{O}/^{16}\text{O}$ ratio of water from vinegar is determined on CO_2 gas after equilibration of reference CO_2 gas with vinegar water according to the following isotopic exchange reaction:



After equilibration the carbon dioxide in the gaseous phase is used for analysis by means of Isotopic Ratio Mass Spectrometry (IRMS) where the $^{18}\text{O}/^{16}\text{O}$ isotopic ratio is determined on the CO_2 resulting from the equilibration.

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 Carbon dioxide

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

4.2 Carbon dioxide used for equilibration

Depending on the instrument, this gas could be the same as 4.1 or in the case of continuous flow systems cylinders containing gas mixture helium-carbon dioxide can also be used

5 Apparatus

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

5.1 Vials with septa appropriate for the used system.