

STN	Pôvod potravín Izotopická analýza kyseliny octovej a vody v octe Časť 1: ^2H-NMR analýza kyseliny octovej	STN EN 16466-1 58 1330
------------	--	--

Food authenticity - Isotopic analysis of acetic acid and water in vinegar - Part 1: ^2H -NMR analysis of acetic acid

Táto norma obsahuje anglickú verziu európskej normy.
This standard includes the English version of the European Standard.

Táto norma bola oznámená vo Vestníku ÚNMS SR č. 02/25

Obsahuje: EN 16466-1:2024

Oznámením tejto normy sa ruší
STN EN 16466-1 (58 1330) z apríla 2013

139856



EUROPEAN STANDARD

EN 16466-1

NORME EUROPÉENNE

EUROPÄISCHE NORM

November 2024

ICS 67.220.10

Supersedes EN 16466-1:2013

English Version

Food authenticity - Isotopic analysis of acetic acid and water in vinegar - Part 1: ^2H -NMR analysis of acetic acid

Authenticité des aliments - Analyse isotopique de l'acide acétique et de l'eau dans le vinaigre - Partie 1 : analyse RMN- ^2H de l'acide acétique

Lebensmittelauthentizität - Isotopenanalyse von Essigsäure und Wasser in Essig - Teil 1: ^2H -NMR-Analyse von Essigsäure

This European Standard was approved by CEN on 24 June 2024.

This European Standard was corrected and reissued by the CEN-CENELEC Management Centre on 4 December 2024.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

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.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Contents		Page
European foreword		3
Introduction		4
1	Scope	5
2	Normative references	5
3	Terms and definitions	5
4	Principle	5
5	Reagents	6
6	Apparatus	6
7	Procedure	7
7.1	Extraction of acetic acid from vinegar	7
7.1.1	General	7
7.1.2	Liquid-liquid extraction	7
7.1.3	Purification of the extract	8
7.2	Composite NMR determination of acetic acid from vinegar	10
7.2.1	General	10
7.2.2	NMR preparation	10
7.2.3	Acquisition of composite NMR spectra	10
7.2.4	Calculations and expression of the result	11
7.2.5	Quality control of the analysis	12
8	Precision	12
8.1	General	12
8.2	Repeatability	12
8.3	Reproducibility	12
Annex A (informative) Results of the collaborative study (2020)		13
Bibliography		14

European foreword

This document (EN 16466-1:2024) has been prepared by Technical Committee CEN/TC 460 “Food authenticity”, the secretariat of which is held by DIN.

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 May 2025, and conflicting national standards shall be withdrawn at the latest by May 2025.

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

This document supersedes EN 16466-1:2013.

EN 16466-1:2024 includes the following significant technical changes with respect to EN 16466-1:2013:

- a) extension to matrices other than wine vinegar;
- b) modification of the NMR sequence;
- c) standard editorially revised.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and the United Kingdom.

EN 16466-1:2024 (E)**Introduction**

Vinegar is defined in EN 13188 [3] 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 [4] defines acetic acid as “Product made from materials of non-agricultural origin”.

Wine vinegar is defined by the European Regulations 1308/2013 [5] 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. Similarly, cider vinegar is defined as the product obtained from the acetous fermentation of apple. Thus, the acetic acid part of these vinegars cannot be obtained from the fermentation of exogenous sugars (e.g. from beet or cane).

Council Regulation (EC) No 813/2000 of 17 April 2000 supplementing the Annex to Commission Regulation (EC) No 1107/96 on the registration of geographical indications and designations of origin under the procedure laid down in Article 17 of Regulation (EEC) No 2081/92 [6] defines Aceto Balsamico Tradizionale di Modena and Commission Regulation (EC) No 583/2009 of 3 July 2009 entering a name in the register of protected designations of origin and protected geographical indications [Aceto Balsamico di Modena (PGI)] [7] defines Aceto Balsamico di Modena as PGI.

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 ^2H -NMR and ^{13}C -IRMS enables the distinction of vinegar origin from other sources, such as beet, cane, malt, apple and synthesis compared to grapes [8]. Isotopic methods have been recognized by the European Committee for Standardization (CEN) and in part by the Organization Internationale de la Vigne et du Vin (OIV) as a means of detecting the non-permitted presence of exogenous acetic acid and water in vinegar (CEN) and specifically wine vinegar (OIV). The methods used are EN 16466-1 for D/H in the methyl site of acetic acid $[(\text{D}/\text{H})_{\text{CH}_3}]$ using ^2H -NMR (Site Specific Natural Isotope Fractionation-Nuclear Magnetic Resonance), EN 16466-2 [16] and OIV 510/2013 [18] for analysis of $^{13}\text{C}/^{12}\text{C}$ in acetic acid using IRMS (Isotope Ratio Mass Spectrometry), and EN 16466-3 [17] and OIV 511/2013 [19] for analysis of $^{18}\text{O}/^{16}\text{O}$ in water using IRMS. Recently, it was experimentally proven that OIV and CEN methods are also applicable to the analysis of acetic acid extracted from Aceto Balsamico di Modena (D/H, ^{13}C) [9].

This document provides the base for the analytical methods. The setup of the required apparatus depends to a large extent on its design principles, and the specific recommendations of the manufacturers should be followed. It is intended to serve as a frame in which analysts can define their own analytical work in accordance with the standard procedure.

This document has been based on an international collaborative study [1]; and is an update and extension of the previous version published in 2013 after a first international collaborative study [2], and organized under the auspices of the Permanent International Vinegar Committee (CPIV, Brussels).

The application of this document can involve the use of hazardous substances, operations and equipment. This document does not claim to address all associated safety issues. It is the responsibility of the user of this document to take appropriate measures for the safety and health protection of personnel before use, and to check the applicability of existing national and European rules and regulations.

1 Scope

This document specifies an isotopic method to control the authenticity of vinegar and food containing vinegar as an ingredient (for example Aceto Balsamico di Modena), with a density below 1,28 g/cm³. This method is applicable on acetic acid of vinegar (from wine, cider, agricultural alcohol, etc.) in order to characterize 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 ²H-NMR is based on a similar method already normalized for wine analysis^[10].

2 Normative references

There are no normative references in this document.

koniec náhľadu – text ďalej pokračuje v platenej verzii STN