BS EN 17992:2024

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Food authenticity — Determination of the sum of 16-O-Methylcafestol, 16-O-Methylkahweol and their derivatives in roasted coffee by ^{1}H-qNMR

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee AW/34/46, Food Authenticity.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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Published by BSI Standards Limited 2024

ISBN 978 0 539 27683 1

ICS 67.140.20

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2024.

Amendments/corrigenda issued since publication

Date	Text affected

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 17992

November 2024

ICS 67.140.20

English Version

Food authenticity - Determination of the sum of 16-O-Methylcafestol, 16-O-Methylkahweol and their derivatives in roasted coffee by ^{1}H-qNMR

Authenticité des aliments - Détermination par résonance magnétique nucléaire quantitative du proton (RMN-^{1}H) de la somme des teneurs en 16-O-méthylcafestol, 16-O-méthylkahwéol et leurs dérivés dans le café torréfié

Lebensmittelauthentizität - Bestimmung des Gehalts von 16-O-Methylcafestol, 16-O-Methylkahweol und deren Derivaten als Summenparameter in Röstkaffee mittels ^{1}H-qNMR

This European Standard was approved by CEN on 2 September 2024.

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European foreword

This document (EN 17992:2024) 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of an identical text or by endorsement, at the latest by May 2025, and conflicting national standards shall be withdrawn at the latest by May 2025.

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Introduction

This document was developed in response to standardization demand for an efficient and reliable test method allowing the confirmation of coffee authenticity both for commercial quality control and for official food control.

The coffee species with the greatest commercial importance are *Coffea arabica* and *Coffea canephora var. robusta*, commonly known as "arabica" and "robusta". Within these species, arabica coffees have a significantly higher market value than robusta coffees. For roasted coffee samples the claim "100 % arabica", "pure arabica" can be authenticated by analysing the mass fraction of 16-O-Methylcafestol and 16-O-Methylkahweol and their derivatives (e.g. fatty acid esters). Whereas arabica coffees contain no detectable or only very small amounts of these analytes (less than 20 mg/kg), the mass fractions in robusta coffees are significantly higher in the approximate range of 800 to 2 500 mg/kg [1, [1]-[7]].

Metrologically, quantitative NMR (qNMR) is a proven primary method of measurement [8]. This means that the quantity determination by NMR is directly related to the definition of the unit of measurement and/ or basic fundamental constants. Thus, the measurement process is physically fully understood and a complete error budget and its physical causation can be described. NMR does not need substance specific calibration.

Three approaches to qNMR are common and regularly used:

— use of an internal standard (adding an exact quantity of a reference substance to the sample);

— projection of a concentration-calibrated artificial signal into the sample's spectrum (called ERETIC method) [9];

— calibration of the spectrometer's response (under comparable acquisition conditions) with an external standard of exactly known concentrations (called PULCON method or ERETIC2) [10].

This analysis method uses the PULCON method to quantify analytes with NMR.

[1] These published values are quoted as an orientation only and not intended as strict threshold recommendations.

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1 Scope

This document specifies a method for the determination of soluble 16-O-Methylcafestol and 16-O-Methylkahweol content (the sum of free forms and derivatives, e.g. fatty acid esters, henceforth abbreviated as 16-OMD = "diterpenes") in roasted coffee (beans or ground), using quantitative proton nuclear magnetic resonance spectroscopy (^{1}H-qNMR).

If complying with the experimental parameters described below, this test procedure has been proven for the following mass fraction range:

$w_{16\text{-OMD}}$: 20 mg/kg to 2 000 mg/kg.

The mass fraction range can be expanded by suitable changes of the experimental parameters, e.g. a different weighed portion of ground coffee or the accumulation of more NMR-transients.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 1042, *Laboratory glassware — One-mark volumetric flasks (ISO 1042)*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3509, *Coffee and coffee products — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3509 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp/

— IEC Electropedia: available at https://www.electropedia.org/

3.1
16-OMD content
amount of 16-OMC and 16-OMK (both are 16-O-Methylditerpenes) and their derivatives in coffee

Note 1 to entry: It is extractable with chloroform, determined according to this test procedure and usually quoted as a mass fraction *w* in mg/kg coffee, calculated as free 16-OMC.

Note 2 to entry: An additional fraction of the total 16-OMD content (ca. 25 %) can only be extracted by vigorous chemical processing.

Note 3 to entry: Approximately a mass fraction of 90 % of the 16-OMD is represented by 16-O-Methylcafestol, and 16-O-Methylkahweol represent the remaining mass fraction of 10 % of the mixture.

Note 4 to entry: The relative error due to differences in molar weight is 0,6 %.

[SOURCE: bibliography [1]]

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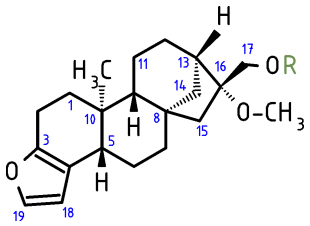
3.2
16-OMC
16-O-Methylcafestol
(CAS RN®: 108214-28-4), M = 330,5 g/mol

Note 1 to entry: The substance appears in its free form or esterified, see Figure 1.



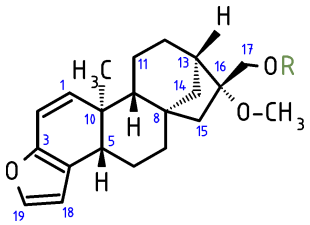
Key

R residue (H or fatty acid)

Figure 1 — 16-OMC

3.3
16-OMK
16-O-Methylkahweol
(CAS RN®: not yet available), M = 328,5 g/mol

Note 1 to entry: The substance appears in its free form or esterified, see Figure 2.



Key

R residue (H or fatty acid)

Figure 2 — 16-OMK

3.4
FID
free induction decay
time-domain NMR signal that results from the precession of the nuclear magnetization vector inside the probe coil after application of an excitation RF (radio frequency) pulse to a sample in a static magnetic field

[SOURCE: ISO 24583:2022, 3.6, modified – RF explanation added]

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3.5
ERETIC
Electronic REferencing To In-vivo Concentrations

quantitative NMR method using a synthetic signal with an integral calibrated against an exactly known concentration of protons

Note 1 to entry: By adding this signal to a sample's real spectrum, this spectrum has an artificial signal that can be used for concentration determination like the signal of an internal standard.

Note 2 to entry: The advantage of the ERETIC signal is that it can be set in an empty region of the sample spectrum, the disadvantage is that an extra signal generating electronic circuitry is needed in the spectrometer.

Note 3 to entry: ERETIC2 is a commercial word mark for the PULCON method.

3.6
FWHM
full width at half maximum
width of a line shape at half the maximum signal intensity

Note 1 to entry: It is expressed in Hz.

[SOURCE: ISO 24583:2022, 3.9 – modified: Note 2 to entry deleted]

3.7
NMR
nuclear magnetic resonance spectroscopy
method based on the selective absorption of high frequency radio waves by atomic nuclei subjected to a stationary magnetic field

Note 1 to entry: NMR provides chemical and structural properties of molecules.

[SOURCE: ISO 23118:2021, 3.10]

3.8
ppm
part per million
<NMR> pseudo-unit for the chemical shift (δ) of a resonance signal

Note 1 to entry: The frequency difference of a signal's resonance f_{signal} from a reference compound's resonance f_{referene} (both frequencies in Hz, cycles per second) is divided by the frequency of the reference compound's resonance and multiplied with 10^6. This yields a normalized shift measure that is independent of the spectrometer's field strength. See the following formula for explanation:

$$\delta_{\text{signal}} = \frac{f_{\text{signal}} - f_{\text{reference}}}{f_{\text{reference}}} \times 10^6$$

Note 2 to entry: ppm is not a (SI) unit but a symbol for an order of magnitude (10–6), like the % symbol (10–2).

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