SLOVENSKI STANDARD
SIST EN ISO 18363-3:2022

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Živalske in rastlinske maščobe ter olja - Določevanje maščobnokislinsko vezanih kloropropandiolov (MCPD) in glicidola z GC/MS - 3. del: Metoda z uporabo kislinske transesterifikacije in meritev 2-MCPD, 3-MCPD in glicidola (ISO 18363-3:2017)

Animal and vegetable fats and oils - Determination of fatty-acid-bound chloropropanediols (MCPDs) and glycidol by GC/MS - Part 3: Method using acid transesterification and measurement for 2-MCPD, 3-MCPD and glycidol (ISO 18363-3:2017)

Tierische und pflanzliche Fette und Öle - Bestimmung von fettsäuregebundenen Chlorpropandiol (MCPD) und Glycidol mittels GC/MS - Teil 3: Verfahren mittels Säureumesterung und Messung für 2 MCPD, 3 MCPD und Glycidol (ISO 18363 3:2017)

Corps gras d'origines animale et végétale - Détermination des esters de chloropropanediols (MCPD) et d'acides gras et des esters de glycidol et d'acides gras par CPG/SM - Partie 3: Méthode par transestérification acide et mesure du 2-MCPD, du 3-MCPD et du glycidol (ISO 18363-3:2017)

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EUROPEAN STANDARD

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EN ISO 18363-3

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ICS 67.200.10

English Version

Animal and vegetable fats and oils - Determination of fatty-acid-bound chloropropanediols (MCPDs) and glycidol by GC/MS - Part 3: Method using acid transesterification and measurement for 2-MCPD, 3-MCPD and glycidol (ISO 18363-3:2017)

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Tierische und pflanzliche Fette und Öle - Bestimmung von fettsäuregebundenen Chlorpropandiol (MCPD) und Glycidol mittels GC/MS - Teil 3: Verfahren mittels Säureumesterung und Messung für 2 MCPD, 3 MCPD und Glycidol (ISO 18363 3:2017)

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European foreword

The text of ISO 18363-3:2017 has been prepared by Technical Committee ISO/TC 34 "Food products" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 18363-3:2021 by Technical Committee CEN/TC 307 "Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis" the secretariat of which is held by AFNOR.

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 June 2022, and conflicting national standards shall be withdrawn at the latest by June 2022.

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Endorsement notice

The text of ISO 18363-3:2017 has been approved by CEN as EN ISO 18363-3:2021 without any modification.

INTERNATIONAL STANDARD

ISO 18363-3

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Animal and vegetable fats and oils — Determination of fatty-acid-bound chloropropanediols (MCPDs) and glycidol by GC/MS —

Part 3: Method using acid transesterification and measurement for 2-MCPD, 3-MCPD and glycidol

Corps gras d'origines animale et végétale — Détermination des esters de chloropropanediols (MCPD) et d'acides gras et des esters de glycidol et d'acides gras par CPG/SM —

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Foreword

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This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

A list of all parts in the ISO 18363 series can be found on the ISO website.

Introduction

The ISO 18363 series is a family of International Standards which can be used for the determination of ester-bound MCPD and glycidol. This introduction describes the methods specified in the three documents currently published or proposed so that the analyst can decide which methods are suitable for application. The detailed application of each method is contained within the scope of the individual method.

ISO 18363-1 is a differential method equivalent to the DGF standard C-VI 18 (10) and identical to AOCS Official Method Cd 29c-13. Briefly, it is based on a fast alkaline catalysed release of 3-MCPD and glycidol from the ester derivatives. Glycidol is subsequently converted into induced 3-MCPD. It consists of two parts. The first part (A) allows the determination of the sum of ester-bound 3-MCPD and ester-bound glycidol, whereas the second part (B) determines ester-bound 3-MCPD only. Both assays are based on the release of the target analytes 3-MCPD and glycidol from the ester-bound form by an alkaline catalysed alcoholysis carried out at room temperature. In part A, an acidified sodium chloride solution is used to stop the reaction and subsequently convert the glycidol into induced 3-MCPD. Thus, 3-MCPD and glycidol become indistinguishable in part A. In part B, the reaction stop is achieved by the addition of an acidified chloride-free salt solution which also prevents the conversion of glycidol into induced MCPD. Thereby, part B allows the determination of the genuine 3-MCPD content. Finally, the glycidol content of the sample is proportional to the difference of both assays (A – B) and can be calculated when the transformation ratio from glycidol to 3-MCPD has been determined. ISO 18363-1 is applicable to the fast determination of ester-bound 3-MCPD and glycidol in refined and non-refined vegetable oils and fats. ISO 18363-1 can also apply to animal fats and used frying oils and fats, but a validation study has to be undertaken before the analysis of these matrices. Any free analytes within the sample would be included in the results, but the document does not allow the distinction between free and bound analytes. However, as of publication, research has not shown any evidence of a free analyte content as high as the esterified analyte content in refined vegetable oils and fats. In principle, ISO 18363-1 can also be modified in such a way that the determination of 2-MCPD is feasible, but again, a validation study has to be undertaken before the analysis of this analyte.

ISO 18363-2 (proposed) will represent AOCS Official Method Cd 29b-13. Briefly, it will be based on a slow alkaline release of MCPD and glycidol from the ester derivatives. Glycidol is subsequently converted into 3-MBPD. ISO 18363-2 will consist of two sample preparations that differ in the use of internal standards. Both preparations will be used for the determination of ester-bound 2-MCPD and 3-MCPD. In part A, a preliminary result for ester-bound glycidol will be determined. Because the 3-MCPD present in the sample will be converted to some minor extent into induced glycidol by the sample preparation, part B will serve to quantify this amount of induced glycidol that is subsequently subtracted from the preliminary glycidol result of part A. By the use of isotopically labelled free MCPD isomers in assay A and isotopically labelled ester-bound 2-MCPD and 3-MCPD in part B, the efficiency of ester cleavage will be able to be monitored. Both assays A and B will be based on the release of the target analytes 2-MCPD, 3-MCPD, and glycidol from the ester-bound form by a slow alkaline catalysed alcoholysis in the cold. In both sample preparations, the reaction will be stopped by the addition of an acidified concentrated sodium bromide solution so as to convert the unstable and volatile glycidol into 3-MBPD which shows comparable properties to 3-MCPD with regard to its stability and chromatographic performance. Moreover, the major excess of bromide ions will prevent the undesired formation of 3-MCPD from glycidol in the case of samples which contain naturally occurring amounts of chloride. ISO 18363-2 will be applicable to the determination of ester-bound 3-MCPD, 2-MCPD, and glycidol in refined and unrefined vegetable oils and fats. It will also apply to animal fats and used frying oils and fats, but a validation study will have to be undertaken before the analysis of these matrices. Any free analytes within the sample would be included in the results, but the document will not allow the distinction between free and bound analytes. However, as of publication of this document, research has not shown any evidence of a free analyte content as high as the esterified analyte content in vegetable oils and fats.

This document represents AOCS Official Method Cd 29a-13. Briefly, it is based on the conversion of glycidyl esters into 3-MBPD esters and a slow acid catalysed release of MCPD and MBPD from the ester derivatives. This document is based on a single sample preparation in which glycidyl esters are converted into MBPD monoesters, and subsequently, the free analytes 2-MCPD, 3-MCPD, and 3-MBPD are released by a slow acid catalysed alcoholysis. The 3-MBPD represents the genuine content of bound glycidol. This document can be applied for the determination of ester-bound 2-MCPD, 3-MCPD, and

glycidol in refined and non-refined vegetable oils and fats. It can also apply to animal fats and used frying oils and fats, but a validation study has to be undertaken before the analysis of these matrices. The method is suited for the analysis of bound (esterified) analytes, but if required, this document can be also performed without the initial conversion of glycidyl esters. In such a setup, both free and bound 2-MCPD and 3-MCPD forms would be included in the results and the amount of free analytes can be calculated as a difference between two determinations performed in both setups. However, as of publication of this document, research has not shown any evidence of a free analyte content as high as the esterified analyte content in vegetable oils and fats.