INTERNATIONAL STANDARD

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

Part 1: Method using fast alkaline transesterification and measurement for 3-MCPD and differential measurement for 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 —

Partie 1: Méthode par transestérification alcaline rapide et mesure pour le chloro-3 propane-1,2-diol (3-MCPD) et par mesure différentielle pour le glycidol

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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

ISO 18363 consists of the following parts, under the general title *Animal and vegetable fats and oils — Determination of fatty-acid-bound chloropropanediols (MCPDs) and glycidol by GC/MS*

— *Part 1: Method using fast alkaline transesterification and measurement for 3-MCPD and differential measurement for glycidol*

The following parts are under preparation:

— *Part 2: Method using alkaline transesterification and measurement for 2-MCPD, 3-MCPD and glycidol*

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

Introduction

ISO 18363 is a set of International Standards which can be used for the determination of ester-bound MCPD and glycidol. There are currently three International Standards which have been proposed and this introduction is a description of these methods, which can be used by the analyst to decide which methods are suitable for their application. The detailed application of each method is contained within the scope of the individual method.

This part of ISO 18363 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. This part of ISO 18363 is applicable for the fast determination of ester bound 3-MCPD and glycidol in refined and non-refined vegetable oils and fats. This part of ISO 18363 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 standard 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, this part of ISO 18363 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.

The second part of the proposed International Standards for the determination of ester-bound MCPD and glycidol represents the AOCS Official Method Cd 29b-13. Briefly, it is based on a slow alkaline release of MCPD and glycidol from the ester derivatives. Glycidol is subsequently converted into 3-MBPD. The second part of the proposed International Standards consists of two sample preparations that differ in the use of internal standards. Both parts can be used for the determination of ester bound 2-MCPD and 3-MCPD. In part A, a preliminary result for ester bound glycidol is 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 serves 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 can be monitored. Both assays A and B are 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 is 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 prevents the undesired formation of 3-MCPD from glycidol in the case of samples which contain naturally occurring amounts of chloride. The second part of the proposed standards is applicable for the determination of ester bound 3-MCPD, 2-MCPD, and glycidol in refined and unrefined vegetable oils and fats. The second part of the proposed International Standards 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 standard 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 vegetable oils and fats.

The third part of the proposed International Standards for the determination of ester-bound MCPD and glycidol represents the AOCS Official Method Cd 29a-13. Briefly, it is based on the conversion of glycidyl esters into 3-MBPD esters and a slow acidic catalysed release of MCPD and MBPD from the