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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 44621-2024

**Inspection of grain and oils - Determination of fatty-acid-bound
3-chloropropane-1,2-diols (MCPDs) and glycidol by GC/MS**

粮油检验 GC/MS法测定3-氯丙醇脂肪酸酯和缩水甘油脂肪酸酯

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Foreword

This document is in accordance with the provisions of GB/T 1.1-2020 "Guidelines for standardization work Part

1. Structure and drafting rules for standardization documents" Drafting. This document is modified to adopt ISO 18363-1.2015 "Determination of monochloropropanol fatty acid esters and glycidol in animal and vegetable oils and fats by GC/MS" Fatty acid esters - Part

1. Rapid alkaline transesterification for the determination of 3-MCPD and glycidol by differential method", ISO 18363-2.2018 Determination of monochloropropanol fatty acid esters and glycidyl fatty acid esters by GC/MS in animal and vegetable oils and fats Part

2. Slow alkaline transesterification ISO 18363-3.2017 "Determination of monochloropropanol in animal and vegetable oils and fats by GC/MS" Fatty acid esters and glycidyl fatty acid esters Part

3. Acid transesterification for the determination of 2-chloropropanol, 3-chloropropanol and glycidol ISO 18363-4.2021 "Determination of monochloropropanol fatty acid esters and glycidyl fatty acid esters in animal and vegetable fats and oils by GC/MS - Part 4. Rapid alkaline transesterification for the determination of 2-chloropropanol, 3-chloropropanol and glycidol. This document is compared with ISO 18363-1.2015, ISO 18363-2.2018, ISO 18363-3.2017 and ISO 18363-4.2021. There are many structural adjustments. For a comparison of the structural number changes between each two files, see Table A.1 to Table A.4 in Appendix A. Compared with ISO 18363-1.2015, ISO 18363-2.2018, ISO 18363-3.2017 and ISO 18363-4.2021, this document has In the case of more technical differences, a single vertical line (|) is used to mark the blank space outside the relevant

clause. For a list of reasons, see Tables A.5 to A.8 in Appendix A. The following editorial changes were made to this document.

--- Changed the standard name;

--- Deleted the notes of international standards. Please note that some of the contents of this document may involve patents. The issuing organization of this document does not assume the responsibility for identifying patents. This document was proposed by the State Administration of Grain and Material Reserves. This document is under the jurisdiction of the National Technical Committee on Cereals and Oils Standardization (SAC/TC270). This document was drafted by: National Grain and Material Reserves Administration Scientific Research Institute, National Grain and Material Reserves Administration Standard Quality Center, South China University of Science and Technology, Nanjing Customs Animal and Plant and Food Inspection Center, Henan University of Technology, Sinograin Zhenjiang Quality Inspection Center Co., Ltd., Shaanxi Keyiyang Optical Detection Technology Service Co., Ltd., Wilmar (Shanghai) Biotechnology Research and Development Center Co., Ltd., Shandong Xingquan Oil Co., Ltd., Qingdao Tianxiang Xiyan Food Co., Ltd., Yunnan Dianxue Cereals and Oils Co., Ltd., COFCO Nutrition and Health Research Institute Co., Ltd., COFCO Engineering (Xi'an) International Engineering Co., Ltd., Shandong Jinsheng Cereals, Oils and Food Co., Ltd., Shandong Longda Cereals and Oils Co., Ltd., Feixian COFCO Oils and Fats Industry Co., Ltd., Zhengzhou PONY Testing Technology Co., Ltd., Quzhou Liujiaxiang Food Co., Ltd. The main drafters of this document are. Duan Zhangqun, Yang Kaizhou, Wei Zheng, Zhang Yan, Shen Weijian, Xue Yalin, Liu Guoqin, Chen Xiaowei, Bi Yanlan, Zhang Liuping, Luo Shilong, Wang Meng, Wang Jiaya, Li Yanyan, Li Zisong, Yu Qiang, Zeng Xiangju, Wang Xiangyu, Zhang Xiaoyong, Qi Xiaozhe, Sun Qincal, Gao Hongliang, Liu Peilian, Li Yingfang and Liu Jingwei. Grain and Oil Inspection-GC/MS Determination of 3-chloropropanol Fatty acid esters and glycidyl fatty acid esters

1 Scope

GB/T 44621-2024 gives the Chinese GC/MS method for determining fatty acid esters of 3-monochloropropane-1,2-diol and of glycidol in edible oils and fats. These are process contaminants: they are not present in the raw oil but are formed during the high temperature deodorisation step of refining, and because glycidyl esters are hydrolysed to glycidol in the gut and 3-MCPD esters to 3-MCPD, both are regulated as genotoxic or nephrotoxic concerns, with infant formula fat the most sensitive application. The analysis is difficult and famously method-dependent, which is why a national method matters: the esters cannot be measured directly, so the procedure transesterifies them, derivatises the released diols and quantifies by GC/MS, and every choice in that chain, the alkaline or acidic route, the reaction time, the derivatising agent, changes the answer. The standard sets the principle, reagents, apparatus, sample preparation, the transesterification and

derivatisation procedure, the chromatographic and mass spectrometric conditions, the calibration and calculation, and the precision. Informative annexes give the technical differences from the international method, the interlaboratory verification results of the difference approach and the reference chromatograms. It takes effect on 1 April 2025.

This document describes the method for the determination of 3-chloropropanol fatty acid esters and glycidyl fatty acid esters, including the test principle, reagents and materials, instrumentation, and Instruments and equipment, sample preparation, test procedures, result calculation, precision, etc. This document applies to the determination of 3-chloropropanol fatty acid esters and glycidyl fatty acid esters in solid and liquid oils. It is used for the determination of 3-chloropropanol fatty acid esters and glycidyl fatty acid esters in oils and fats. Chapters 5, 6 and 7 are applicable to the determination of 3-chloropropanol fatty acid esters and glycidyl fatty acid esters in oils and fats. Determination of 2-propanol fatty acid esters, 2-chloropropanol fatty acid esters and glycidyl fatty acid esters. Chapter 5 does not apply to milk and milk products and their fats. Chapter 7 does not apply to milk and dairy products and their fats, infant formula, emulsifiers, free fatty acids and other oil derivatives. The limit of detection (LOD) of this document is 30µg/kg and the limit of quantification (LOQ) is 100µg/kg.

2 Normative references

The contents of the following documents constitute essential clauses of this document through normative references in this document. For referenced documents without a date, only the version corresponding to that date applies to this document; for referenced documents without a date, the latest version (including all amendments) applies to This document.

GB/T 6682 Specifications and test methods for water used in analytical laboratories (GB/T 6682-2008, ISO 3696.1987, MOD)1) 1)

ISO 3696.1987 has been superseded by ISO 3696.2021.

GB/T 15687 Preparation of animal and vegetable fats and oils samples (GB/T 15687-2008, ISO 661.2003, IDT)

3 Terms and definitions

There are no terms or definitions that require definition in this document.

4 Rapid alkaline transesterification for determination of 3-MCPD and differential determination of glycidol (referred to as differential method)

4.1 Principle This method consists of two experimental parts. After base-catalyzed ester cleavage, 3-chloropropanol fatty acid ester and glycidol fatty acid ester are respectively In test (A), an acidic aqueous solution of sodium chloride was added to convert glycidol into

3-chloropropanol. In the test (B), an acidic aqueous solution of sodium bromide was added to convert glycidol into 3-bromopropanol; after derivatization with phenylboronic acid, a gas chromatography-mass spectrometry (GC-MS) was performed. Spectral analysis, internal standard method for quantification; Test (A) 3-chloropropanol content is used to express the content of 3-chloropropanol fatty acid esters and glycidyl fatty acid esters in the sample. The content of 3-chloropropanol fatty acid ester in the sample is expressed as 3-chloropropanol; the content of glycidyl fatty acid ester in the sample is expressed as the sum of the two. The content of glycidyl fatty acid ester was calculated from the conversion rate of glycidol to 3-chloropropanol, which was proportional to the difference (AB) of some tests.

4.2 Reagents and materials Unless otherwise specified, all reagents used are analytically pure reagents; the test water should comply with the requirements of Grade 1 water in GB/T 6682.

4.2.1 Toluene. chromatographic grade.