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

GB/T 44966-2024

**Determination of the content of fatty acid ethyl esters in olive
oil - Gas chromatography-mass spectrometry**

橄榄油中脂肪酸乙酯含量的测定 气相色谱-质谱法

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Table of Contents

- 1 Scope
- 2 Normative references
- 3 Terms and definitions
- 4 Principle
- 5 Test conditions
- 5.1 Chromatographic conditions

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. 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: Nanjing Customs Animal and Plant and Food Inspection Center, National Grain and Material Reserves Administration Standard Quality Center, National Grain Food and Material Reserves Bureau Scientific Research Institute, Zhejiang Academy of Agricultural Sciences, Shanghai Anpu Experimental Technology Co., Ltd., Nanjing University of Science and Technology, China Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences. The main drafters of this document are: Shen Weijian, Lu Huiyuan, Duan Zhangqun, Zhang Hu, Wang Lijuan, Zhang Yan, Wang Hong, Tang Yongxing, Huang Xinyi, Zhang Chizhong, Zhu Jingyi, Pei Dong. Determination of fatty acid ethyl ester content in olive oil Gas chromatography-mass spectrometry

1 Scope

GB/T 44966-2024 gives the Chinese GC-MS method for determining fatty acid ethyl esters in olive oil. The measurement exists for one purpose: fatty acid ethyl esters form when poor-quality or fermented olives are processed, and their content is the single most effective indicator that an oil sold as extra virgin has been deodorised or blended with lower-grade material, which is why it became the key authenticity parameter in the international trade. China is a large and growing importer of olive oil and needed its own method to enforce the claim. The standard sets the principle, the reagents and apparatus,

the preparation of the sample and the isolation of the ester fraction by chromatography on silica, the gas chromatographic separation, the mass spectrometric detection with the ions monitored for each ester, the identification and the quantification against an internal standard, the calculation and expression of the result, the precision and the test report. It takes effect on 1 June 2025.

This document describes a method for the determination of fatty acid ethyl esters in olive oil using gas chromatography-mass spectrometry. This document applies to the determination of fatty acid ethyl ester content in olive oil.

2 Normative references

The contents of the following documents constitute the 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

3 Terms and definitions

The following terms and definitions apply to this document.

3.1 Esters of higher fatty acids and ethanol.

Note. Includes ethyl palmitate, ethyl stearate, ethyl oleate and ethyl linoleate. [Source: GB/T 23347-2021, 3.12]

4 Principle

The fatty acid ethyl ester in the sample was dissolved in n-hexane, purified by silica gel solid phase extraction column, and analyzed by gas chromatography-mass spectrometry. Quantitative.

5.1 Chromatographic conditions

5.1.1 Carrier gas flow rate. 1 mL/min.

5.1.2 Inlet temperature. 300°C.

5.1.3 Injection mode. non-split injection, split valve opening time is

5.1.4 Carrier gas. helium (purity $\geq 99.999\%$).

5.1.5 Chromatographic column. quartz capillary column for mass spectrometry, 30m (length) x 0.25mm (inner diameter) x 0.25 μ m (film thickness), stationary phase is 5%

Phenyl-95% methyl polysiloxane, or equivalent.

5.1.6 Column temperature. Initial temperature 150°C, increase to 200°C at 20°C/min, increase to 240°C at 2.5°C/min, maintain for 1.5min, Increase the temperature at 35°C/min to 310°C and hold for 2 min.

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