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

GB/T 46666-2025

**Inspection of grain and oils - Determination of squalene in
vegetable oils - Gas chromatographic method**

粮油检验 植物油中角鲨烯的测定 气相色谱法

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part

1. Structure and Drafting Rules of Standardization Documents". Drafting. Please note that some content in this document may involve patents. The issuing organization of this document assumes no responsibility for identifying patents. This document was proposed by the National Food and Strategic Reserves Administration. This document is under the jurisdiction of the National Technical Committee on Standardization of Grain and Oil (SAC/TC270). This document was drafted by: the National Food and Strategic Reserves Administration Research Institute, Ganzhou Comprehensive Inspection and Testing Institute, and Jia Ge Foods (China) Co., Ltd. Limited Company, Beijing Nutrition Source Research Institute Co., Ltd., China Certification & Inspection Group Shandong Testing Co., Ltd., Shandong Provincial Grain and Oil Testing Center China Grain Reserves Corporation Zhenjiang Quality Inspection Center Co., Ltd., Guangxi Zhuang Autonomous Region Grain and Oil Quality Inspection Center, Jiangxi Provincial Institute of Inspection, Testing and Certification Food Inspection Testing and Research Institute. The main drafters of this document are. Zhu Lin, Zhang Rui, Wang Songshan, Guo Baoyuan, Xue Yalin, Liu Xianbiao, Wu Xiangqian, Zhang Bing, Cui Yajuan, and Wang Penggong. Dong Bin, Zhang Yingxia, Nong Ziming, Peng Zhibing, Chen Yuan, Zhang Dongsheng. Determination of Squalene in Vegetable Oils in Grain and Oil Testing Gas chromatography

1 Scope

GB/T 46666-2025 is the Chinese national standard covering measuring squalene in vegetable oil by gas chromatography - the saponification and the unsaponifiable fraction, the internal standard, and the quantification of a compound that is both a quality marker of olive oil and a valuable extract in its own right. First edition, under the National Food and Strategic Reserves Administration. In force from 1 May 2026. Issued on 31 October 2025, it

has been in force since 1 May 2026.

This document describes a gas chromatography method for determining the squalene content in vegetable oils. This document applies to the determination of squalene content in vegetable oils.

2 Normative references

The contents of the following documents, through normative references within the text, constitute essential provisions of this document. Dated citations are not included. For references to documents, only the version corresponding to that date applies to this document; for undated references, the latest version (including all amendments) applies. This document.

GB/T 15687 Preparation of animal and vegetable oil samples

3 Terms and Definitions

This document does not contain any terms or definitions that need to be defined.

4.Principles After saponification with potassium hydroxide-ethanol solution and extraction of squalene with n-hexane, the sample was analyzed by gas chromatography with squalane as an internal standard. The content of squalene in the medium.

5.Reagents and Materials Unless otherwise specified, only reagents confirmed to be of analytical grade and distilled or deionized water or water of equivalent purity shall be used in the analysis.

5.1 n-Hexane (C₆H₁₄). chromatographic grade.

5.2 Potassium hydroxide-ethanol solution (approximately 1 mol/L). Weigh 60 g ±

0.1 g potassium hydroxide, dissolve in 50 mL of water, cool to room temperature, and then add... Add 950 mL of 95% (volume fraction) ethanol, stir well, and transfer to a reagent bottle with a rubber stopper. The solution should be colorless or pale yellow. 5.3 10% ethanol solution. Measure 50 mL of anhydrous ethanol into a 500 mL volumetric flask using a graduated cylinder, and dilute to the mark with water.

5.4 Anhydrous sodium sulfate (Na₂SO₄).

5.5 Squalane standard (C₃₀H₆₂, CAS No.. 111-01-3). purity ≥98%, or a standard sample/standard substance with a certificate.

5.6 Squalene standard (C₃₀H₅₀, CAS No.. 111-02-4). purity ≥98%, or standard sample/standard substance with certificate.

5.7 Squalane internal standard solution. Accurately weigh 25 mg (accurate to 0.0001

g) of squalane standard (5.5) into a 25 mL volumetric flask, add... Dissolve a small amount of n-hexane (5.1) completely, then dilute to the mark with n-hexane (5.1), shake well, and a 1 mg/mL squalane internal standard solution is prepared. Liquid. Store sealed and protected from light at -20°C. Shelf life is 3 months. Allow to reach room temperature before use.

5.8 Squalene Standard Solution. Accurately weigh 10 mg (accurate to 0.0001

g) of squalene standard (5.6) into a 50 mL volumetric flask, add... Dissolve a small amount of n-hexane (5.1) completely, then dilute to the mark with n-hexane (5.1), and shake well to prepare a 200 µg/mL squalene standard. Solution. Store sealed and protected from light at -20°C. Shelf life is 3 months. Allow to reach room temperature before use.