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Report Name: National Food Safety Standard of Food Additive Lutein Notified to WTO

Country: China - People's Republic of

Post: Beijing

Report Category: FAIRS Subject Report, Sanitary/Phytosanitary/Food Safety, WTO Notifications

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Report Highlights:

On March 6, 2023, China notified an updated National Food Safety Standard of Food Additive Lutein to the World Trade Organization (WTO) under G/SPS/N/CHN/1266. The deadline for comment submission is May 5, 2023. The proposed date of entry into force is to be determined. Comments may be submitted by email to China's SPS Enquiry Point at sps@customs.gov.cn. This report provides an unofficial translation of the draft standard.

Summary:

On March 6, 2023, China notified an updated National Food Safety Standard of Food Additive Lutein to the WTO under G/SPS/N/CHN/1266. This standard applies to the food additive lutein made from oleoresin of Tagetes erecta L. used as raw material and processed by saponification, extraction, refinement, or other methods. It specifies the technical requirements and testing methods for the food additive lutein. The notified standard is an update of the current National Food Safety Standard of Food Additive Lutein (GB 26405-2011) (link in Chinese), which went into effect in May 2011. Compared with the current standard, the notified standard changes the relative molecular mass, revises the testing methods for lead and total arsenic, and adds Appendices B and C to include gas chromatograms. This report provides an unofficial translation of the draft standard.

BEGIN TRANSLATION

National Food Safety Standard
Food Additive Lutein
(Draft for comments)

Foreword

This standard replaces GB 26405-2011 National Food Safety Standard for Food Additives Lutein. Compared with GB 26405-2011, the main changes in this standard are as follows:

- Modified the scope of commercialized lutein products in the scope,
- Modified the relative molecular weight,
- Modified the status and smell in sensory requirements, and modified the test methods for lead and total arsenic in physical and chemical indicators,
- Modified some descriptions in Appendix A and added purity requirements for reagents,
- Added Appendix B and Appendix C.

National Food Safety Standard
Food Additive Lutein

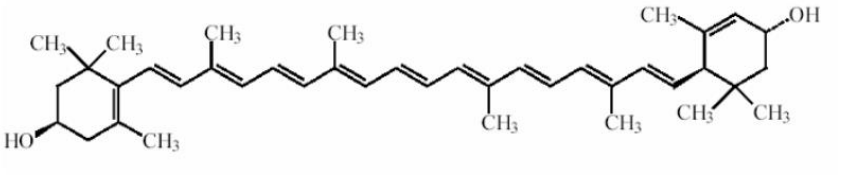
1. Scope

This standard is applicable to the food additive lutein made from Tagetes erecta L. oleoresin through extraction, saponification, refining, and other processes.

2. Molecular Formula, Structural Formula, and Relative Molecular Mass

2.1 Molecular formula
$C_{40}H_{56}O_2$

2.2 Structural formula



2.3 Relative molecular mass
568.85 (according to 2018 international relative atomic mass)

3. Technical Requirements

3.1 Sensory requirements
The sensory requirements shall comply with the provisions of Table 1.

Table 1: Sensory requirements

Item	Requirements	Test Methods
Color	Orange yellow to orange red	Take an appropriate amount of sample and place it on a clean and dry white porcelain plate. Under natural light, observe its color, state, and smell.
Status	Powder	
Odor	It has proper odor, no abnormal smell.	

3.2 Physical and Chemical Indicators
Physical and chemical indicators shall comply with provisions in Table 2.

Table 2: Physical and Chemical Indicators

Item		**Index**	**Testing Methods**
Total carotenoids, w/%	≤	80.0	A.3 in appendix A
Lutein, w/%	≤	70.0	A.4 in appendix A
Zeaxanthin, w/%	≥	9.0	A.4 in appendix A
Drying weight loss, w /%	≥	1.0	GB 5009.3 Reduced Pressure Drying
Ash, w/%	≥	1.0	GB 5009.4
N-hexane/(mg/kg)	≥	50	A.5 in appendix A
Lead (Pb)/(mg/kg)	≥	3.0	GB 5009.12 or GB 5009.75
Total arsenic (in As)/ (mg/kg)	≥	3.0	GB 5009.11 or GB 5009.76
Note: Commercialized lutein products should be made from lutein conforming to this standard, which can be added with food ingredients such as edible vegetable oil, dextrin, sugar, starch, starch sugar, and other food additives that meet the quality specifications for food additives. The state of the products can be powder, particle, suspension, paste, or plaster. The total carotenoids and lutein content should meet the marked values.			

Appendix A
Testing Methods

A.1 General provisions
The reagents and water used in this standard, unless otherwise specified, refer to analytical pure reagents and level I water specified in GB/T 6682. When the solvent used in the test is not specified, it refers to aqueous solution.

A.2 Identification test
A.2.1 It is insoluble in water, slightly soluble in n-hexane, and soluble in ethanol and chloroform.

A.2.2 In the test for determining the total carotenoid content, the sample solution has the maximum absorption near the wavelength of 446 nm.

A.2.3 In the test for determining the lutein content, the retention time of lutein in the sample solution should be the same as that in the standard solution.

A.3 Determination of total carotenoids

A.3.1 Reagents and materials

A.3.1.1 n-hexane

A.3.1.2 Acetone

A.3.1.3 Toluene

A.3.1.4 Anhydrous ethanol

A.3.1.5 Mixed solvent: n-hexane, acetone, toluene, and anhydrous ethanol are mixed in a volume ratio of 10:7:7:6.

A.3.2 Instruments and equipment

A.3.2.1 Analytical balance with a sensitivity of 0.1 mg

A.3.2.2 UV - vis spectrophotometer.

A.3.3 Analysis steps
Weigh 0.03 g to 0.05 g of sample, accurate to 0.0001 g, and place in a 100 mL volumetric flask, dissolve with A3.1.5 mixed solvent to a constant volume and shake well. Accurately transfer 1 mL into a 100 mL volumetric flask, dilute to volume with anhydrous ethanol, and prepare a test solution for testing. Place the test solution in a 1 cm cuvette, use anhydrous ethanol as a blank control, and measure the absorbance at the maximum absorption wavelength at 446 nm ± 1 nm on the UV vis spectrophotometer. (By adjusting the concentration of the sample solution, the absorbance is controlled between 0.3 and 0.7.)

A.3.4 Calculation of results

The content of total carotenoids is calculated by the mass fraction of total carotenoids W_0, and the value is expressed in %, which shall be calculated according to formula (A.1):

$$w_0 = \frac{A}{m} \times V \times \frac{1}{2550} \quad \text{(A.1)}$$

A - Actual measurement of the absorbance of the sample solution,
m - mass of the tested sample, in grams (g),
V - Constant volume of the tested sample solution, in milliliters (mL),
2,550 - The absorption coefficient of 1% of sample solution in anhydrous ethanol at a wavelength of 446 nm.

The experimental results are based on the arithmetic mean of the parallel measurement results. The absolute difference between the two independent measurement results obtained under repeatability conditions shall not be greater than 1.5% of the arithmetic mean, and the calculated results shall be expressed in tenths/. x.

A.4 Determination of lutein and zeaxanthin

A.4.1 Reagents and materials

A.4.1.1 n-hexane: chromatographically pure.

A.4.1.2 Ethyl acetate: chromatographically pure.

A.4.1.3 Lutein standard: purity is more than 80%, stored according to labeled storage conditions.

A.4.1.4 Zeaxanthin standard: purity is more than 80%, stored according to labeled storage conditions.

A.4.2 Instruments and equipment

A.4.2.1 Analytical balance, sensitivity 0.1 mg.

A.4.2.2 High performance liquid chromatography (UV detector, with a wavelength of 446 nm).

A.4.3 Reference chromatographic conditions

A.4.3.1 Chromatographic column: silica gel column, 4.6 mmx250 mm, particle size 3 μm or other equivalent chromatographic column.

A.4.3.2 Mobile phase: prepared as n-hexane: ethyl acetate=70:30 (volume ratio), mixed evenly, then filter with a 0.45 μm of membrane, and degas by ultrasound.

A.4.3.3 Column temperature: room temperature.

A.4.3.4 Mobile phase flow rate: 1.5 mL/min.

A.4.3.5 Sample size: 10 μL.

A.4.4 Analysis steps

A.4.4.1 Preparation of standard solution
Weigh 0.01 g of lutein and zeaxanthin standard products respectively, accurate to 0.0001 g, and place them in a 50 mL volumetric flask. Dissolve them with mobile phase to the constant volume and shake well. Prepare on site as needed.

A.4.4.2 Preparation of sample solution
Weigh 0.02 g to 0.05 g of sample, accurate to 0.0001 g, and place in a 100 mL volumetric flask, dissolve with mobile phase to a constant volume, and shake well.

A.4.4.3 Determination
Under A4.3 reference chromatographic conditions, determine the standard solutions of lutein and zeaxanthin, and record the chromatograms. The separation degree of lutein and zeaxanthin is required to be at least 1.5 based on the peak value of lutein.

The liquid chromatograms of lutein and zeaxanthin standard solutions are as shown in Figure B.1.

Under A4.3 reference chromatographic conditions, determine the sample solution, qualitatively determine the retention time of the standard, and record the chromatogram. Repeat the experiment twice to obtain the average values of the peak areas of lutein and zeaxanthin as a percentage of the total peak areas.

A.4.5 Calculation of results
The content of lutein is calculated as the mass fraction w_1 of lutein, and the value is expressed in %, which is calculated according to formula (A2):

$$w_1 = w_0 \times P_1 \quad \text{(A.2)}$$

The content of zeaxanthin is calculated as the mass fraction w_2 of zeaxanthin, and the value is expressed in %, which is calculated according to formula (A.3):

$$w_2 = w_0 \times P_2 \quad \text{(A.3)}$$

Where:

w_0 - The mass fraction of total carotenoids measured in accordance with A.3 of this standard, expressed in %,
P_1 - Average value of the percentage of lutein peak area in the total peak area,
P_2 - Average value of the percentage of zeaxanthin peak area in the total peak area.

The experimental results are based on the arithmetic mean of the parallel measurement results.

The absolute difference between the two independent measurement results obtained under repeatability conditions shall not be greater than 10 % of the arithmetic mean, and the calculated results shall be expressed in tenths/. x.

A.5 Determination of n-hexane

A.5.1 Reagents and materials

A.5.1.1 n-hexane: chromatography pure.

A.5.1.2 N, N-dimethylformamide (DMF): chromatography pure.

A.5.2 Instruments and equipment

A.5.2.1 Analytical balance with a sensitivity of 0.1 mg.

A.5.2.2 Gas chromatograph with hydrogen flame ion detector (FID) and headspace injector.

A.5.3 Reference chromatographic conditions

A.5.3.1 Chromatographic column: capillary column (Φ 0.53 mm x 30 m), with a fixed phase of 6% of cyanopropyl benzene and 94% of dimethylsiloxane, and a membrane thickness of 3.0 μm, or other equivalent chromatographic column.

A.5.3.2 Carrier gas: nitrogen.

A.5.3.3 Carrier gas flow rate: 3.0 mL/min.

A.5.3.4 Injection port temperature: 220℃.

A.5.3.5 Programmed temperature rise: see Table A.1.

Table A.1: Programmed Temperature Rise

Heating rate ℃/min	Temperature ℃	Duration min
-	40	3
3.5	65	-
20	220	5

A.5.3.6 Detector temperature: 235℃.

A.5.3.7 Injection volume: 1 mL, quantitative ring.

A.5.3.8 Split ratio: 3:1.

A.5.4 Reference Headspace Conditions

A.5.4.1 Equilibrium temperature: 80℃.

A.5.4.2 Balance time: 40.0 min.

A.5.4.3 Quantitative ring temperature: 85℃.

A.5.4.4 Quantitative ring volume: 1 mL.

A.5.4.5 Transmission line temperature: 100℃.

A.5.5 Analysis steps

A.5.5.1 Preparation of reference solution
Take 0.1g (accurate to 0.1 mg) of n-hexane into a 100 mL volumetric flask with 30 mL DMF added in advance, dilute to scale volume with DMF, and shake well. Pipette 0.2 mL of the above solution into a 50 mL volumetric flask and dilute with DMF to the scale. This is the reference solution (the concentration of n-hexane in the reference solution is 0.004 mg/mL).

A.5.5.2 Preparation of sample solution
Take 0.6 g (accurate to 0.1 mg) of the sample, place it in a 20 mL headspace bottle, dilute with 6.0 mL DMF, shake well, and seal with a lid.

A.5.5.3 Determination of control solution and sample solution
Accurately pipette 6.0 mL of the above reference solution in A5.5.1 and place it in a 20 mL headspace bottle for determination. Meanwhile, measure the sample solution in A5.5.2. The gas chromatographic diagram of n-hexane control solution is as shown in Figure C.1.

A.5.6 Calculation of results
The residual amount W_3 of n-hexane is expressed in milligrams per kilogram (mg/kg) and calculated according to Formula (A.4):

$$w_3 = \frac{A_X}{A_R} \times \frac{C_R}{m} \times V \times 10^6 \quad \text{(A.4)}$$

A_X - Peak area of n-hexane in the sample solution,
C_R - concentration of n-hexane in the control solution, in milligrams per milliliter (mg/mL),
A_R - peak area of n-hexane in the control solution,
m - mass of the sample, in milligrams (mg),
V - Constant volume of the sample, in milliliters (mL),
10^6 - Conversion factor.

The experimental results are based on the arithmetic mean of the parallel measurement results. The absolute difference between the two independent measurement results obtained under

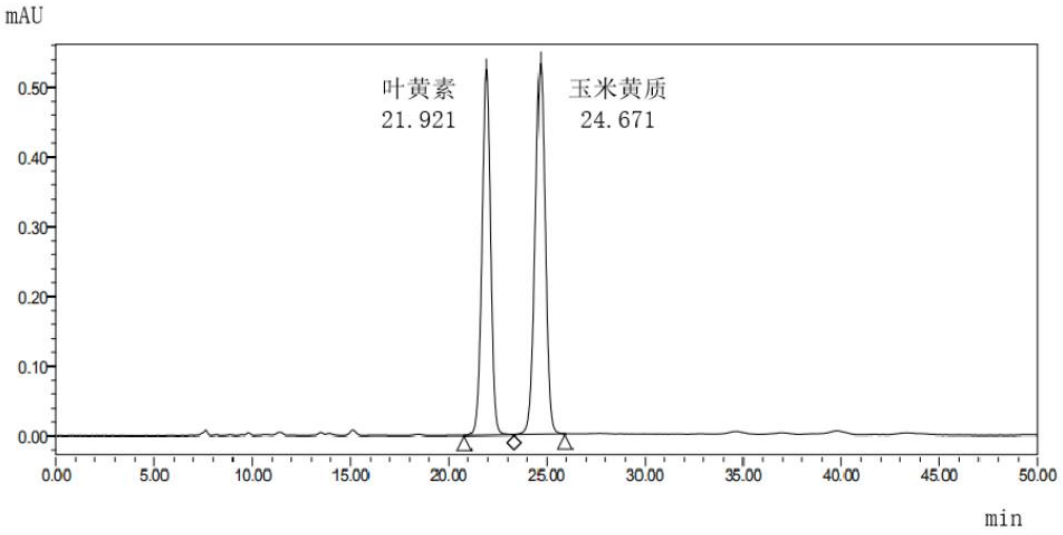
repeatability conditions shall not be greater than 10 % of the arithmetic mean, and the calculated results shall be expressed in tenths/. x.

The detection limit of this method is 2 mg/kg, and the quantitative limit is 10 mg/kg.

Appendix B
The liquid chromatograms of lutein and zeaxanthin standard solutions

The liquid chromatograms of lutein and zeaxanthin standard solutions are as shown in Figure B.1.

Figure B.1: Liquid chromatogram of lutein and zeaxanthin standard solutions

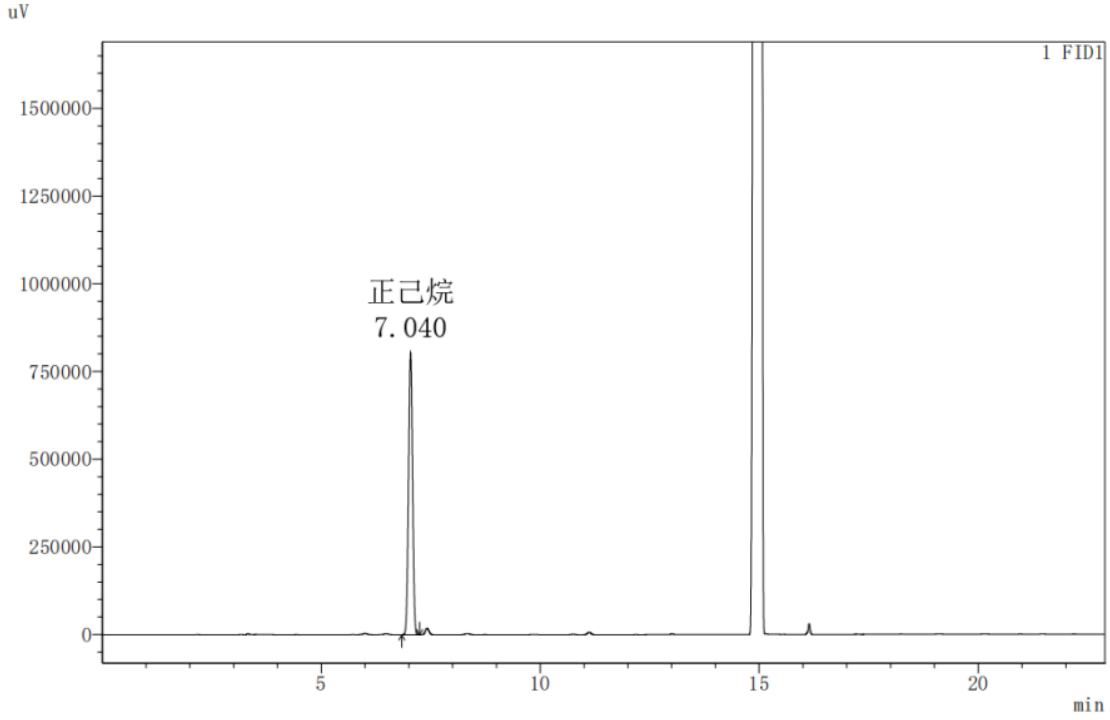


叶黄素: Xanthophyll
玉米黄质: Zeaxanthin

Appendix C
Gas chromatogram of n-hexane reference solution

The gas chromatographic diagram of n-hexane control solution is shown in Figure C.1.

Figure C.1: Gas chromatogram of n-hexane reference solution



正己烷: n-hexane

Attachments:

No Attachments.