

Voluntary Report – Voluntary - Public Distribution

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Report Name: Draft National Food Safety Standard Food Additive Dibutyl Hydroxytoluene 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 July 11, 2024, China notified draft National Food Safety Standard Food Additive Dibutyl Hydroxytoluene (BHT) to the World Trade Organization (WTO) under G/SPS/N/CHN/1306. China's SPS Enquiry Point at sps@customs.gov.cn will accept comments until September 9, 2024. This report provides an unofficial translation of the draft standard. Stakeholders should conduct their own review of the standard and provide comments as necessary.

Report Summary:

On July 11, 2024, China notified draft National Food Safety Standard Food Additive Dibutyl Hydroxytoluene (BHT) to the World Trade Organization (WTO) under [G/SPS/N/CHN/1306](#). China's Sanitary and Phytosanitary (SPS) Enquiry Point at sps@customs.gov.cn will accept comments until September 9, 2024.

This draft standard is applicable to dibutyl hydroxytoluene (BHT), a food additive prepared by distillation and recrystallization of the product of alkylation reaction with p-cresol and isobutylene as raw materials under acidic catalyst. Once finalized, it will replace the current National Food Safety Standard Food Additive Dibutyl Hydroxytoluene (BHT) [GB 1900-2010](#) (link in Chinese).

The draft standard was developed with reference to Codex Alimentarius Commission's standard of butylated hydroxytoluene, formulated at the 30th JECFA (1986), published in FNP 37 (1986), revised at the 37th JECFA (1990), and republished in FNP 52 (1992).

This report provides an unofficial translation of the draft notified standard. Stakeholders should conduct their own review of the standard.

BEGIN TRANSLATION

National Food Safety Standard

**Food Additive Dibutyl Hydroxytoluene (BHT)
(Draft for comments)**

Preface

This standard replaces GB 1900-2010 “Food Additive Dibutyl Hydroxytoluene (BHT).”

Compared with GB 1900-2010, the main changes in this standard are as follows:

- Revised the descriptions for scope;
- Added odor requirements;
- Added main content indicators and testing methods;
- Changed “melting point (initial melting)” to “melting range”;
- Changed “heavy metal (calculated as Pb)” to “lead (Pb)”;
- Modified the identification test.

1. Scope

This standard is applicable to dibutyl hydroxytoluene (BHT), a food additive prepared by distillation and recrystallization of the product of alkylation reaction using p-cresol and isobutylene as raw materials under acidic catalyst (concentrated sulfuric acid, p-toluenesulfonic acid, and sodium p-toluenesulfonate).

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

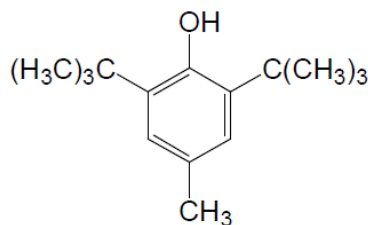
2.1 Chemical name

2,6-di-tert-butyl-p-methylphenol

2.2 Molecular formula

C₁₅H₂₄O

2.3 Structural formula



2.4 Relative molecular mass

220.36 (according to 2022 international relative atomic mass)

3. Technical Requirements

3.1 Sensory requirements

Sensory requirements shall conform to the provisions in Table 1.

Table 1: Sensory Requirements

Items	Requirement	Testing method
Color	White	Take an appropriate amount of the sample and place it on a clean, dry white porcelain plate. Under natural light, observe its color and state, and smell it.
State	Crystalline or crystalline powder	
Smell	Odorless or with a special slight aromatic odor	

3.2 Physical and chemical indicators

It shall conform to the provisions in Table 2.

Table 2: Physical and Chemical Indicators

Items		Indicators	Testing method
Dibutylhydroxytoluene, w/%	≥	99.5	A.4 in Appendix A
Moisture, w/%	≤	0.05	GB 5009.3 Karl Fischer Method ^a
Melting range/°C		69.0~72.0	GB/T 617
Ignition residue, w/%	≤	0.005	GB/T 9741 ^b
Free phenol (calculated as p-cresol), w/%	≤	0.02	A.5 in Appendix A
Sulfate (calculated as SO ₄ ²⁻), w /%	≤	0.002	A.6 in Appendix A
Arsenic (As)/(mg/kg)	≤	1.0	GB 5009.76 or GB 5009.11
Lead (Pb)/(mg/kg)	≤	2.0	GB 5009.75 or GB 5009.12

^a The sample size is approximately 5 g (accurate to 0.0001 g).
^b The sample size is approximately 10 g (accurate to 0.01 g), and the ignition temperature is 800 °C± 25 °C.

Appendix A Testing Method

A.1 Warning

Some of the testing procedures specified in the testing method may lead to dangerous situations. The operator should take appropriate safety and protective measures.

A.2 General Provisions

Unless otherwise specified, only reagents confirmed as analytical grade and grade III water specified in GB/T 6682 shall be used in the analysis.

Unless otherwise specified, the standard titration solution, impurity determination standard solution, preparations and products used in the test method shall be prepared in accordance with the provisions of GB/T 601, GB/T 602, and GB/T 603.

A.3 Identification Test

A.3.1 UV spectrophotometry

A.3.1.1 Reagents and materials

Anhydrous ethanol: guarantee reagent;

A.3.1.2 Instruments and equipment

A.3.1.2.1 UV-visible spectrophotometer: equipped with a 1 cm cuvette.

A.3.1.2.2 Analytical balance: with a sensitivity of 0.0001 g.

A.3.1.3 Analysis steps

A.3.1.3.1 Preparation of sample solution

Weigh approximately 0.1 g of the sample, accurate to 0.0001 g, and place it in a 100 mL volumetric flask. Add an appropriate amount of anhydrous ethanol and sonicate in an ultrasonic water bath for 2 minutes. Shake well, cool, and dilute to a volume of 100 mL solution. Accurately take 1.00 mL of the above solution, place it in a 100 mL volumetric flask, and dilute with anhydrous ethanol to the mark. Then shake well to obtain the sample solution.

A.3.1.3.2 Determination

Using anhydrous ethanol as a reference and a 1 cm cuvette, scan the sample solution within the wavelength range of 230 nm to 320 nm, and there should be maximum absorption at the wavelength of 278 nm.

A.3.2 Gas chromatography

A.3.2.1 Reagents and materials

Dibutyl hydroxytoluene (CAS: 128-37-0, molecular formula: C₁₅H₂₄O) standard product: purity $\geq 99.0\%$.

A.3.2.2 Instruments and equipment

A.3.2.2.1 Gas chromatograph: equipped with hydrogen flame ionization detector.

A.3.2.2.2 Analytical balance: with a sensitivity of 0.0001 g.

A.3.2.3 Analysis steps

Weigh 0.5g of dibutyl hydroxytoluene standard sample (accurate to 0.0001g) into a 10 mL volumetric flask, dissolve in anhydrous ethanol and dilute to the mark, shake well, and obtain the standard solution of dibutyl hydroxytoluene. According to the method specified in A.4, measure the standard solution and sample solution under the same conditions, and record the chromatogram of the sample solution. The retention time of the chromatographic peak of dibutyl hydroxytoluene in the sample solution should be consistent with that of the standard solution, with a relative deviation of within 2.5%. The gas chromatogram of the standard solution of dibutyl hydroxytoluene is as shown in Appendix B.

A.4 Determination of Dibutyl Hydroxytoluene

A.4.1 Method summary

The sample is dissolved in anhydrous ethanol and separated by gas chromatography column under selected working conditions. The components in the sample are detected by flame ionization detector and quantified by area normalization method.

A.4.2 Reagents and materials

A.4.2.1 Anhydrous ethanol: superior level purity

A.4.2.2 Nitrogen: with a volume fraction of not less than 99.99%, dried and purified by silica gel or 5A molecular sieve.

A.4.2.3 Hydrogen: with a volume fraction of not less than 99.99%, dried and purified by silica gel or 5A molecular sieve.

A.4.2.4 Air: Oil free, dried and purified by silica gel or 5A molecular sieve.

A.4.3 Instruments and equipment

A.4.3.1 Gas chromatograph: equipped with hydrogen flame ionization detector.

A.4.3.2 Analytical balance: with a sensitivity of 0.0001 g.

A.4.4 Analysis steps

A.4.4.1 Preparation of sample solution

Weigh about 0.5g of the sample (accurate to 0.0001 g) into a 10 mL volumetric flask, dissolve it in anhydrous ethanol and dilute to volume, and shake well to obtain the sample solution.

A.4.4.2 Operating conditions of gas chromatography

A.4.4.2.1 Chromatographic column: Weak polarity capillary column (bonded, 5% phenyl+95% methyl polysiloxane capillary column), with its length 30 m, inner diameter of 0.32 mm, membrane thickness of 1.00 μ m, or with equivalent performance.

A.4.4.2.2 Detector temperature: 250°C.

A.4.4.2.3 Injection port temperature: 230°C.

A.4.4.2.4 Column temperature: Maintain at 150 °C for 1.0 minute, heat up to 210 °C at 10 °C/min, and maintain for 3 minutes.

A.4.4.2.5 Injection volume: 0.6 μ L.

A.4.4.2.6 Diversion ratio: 1:50.

A.4.4.2.7 Carrier gas: high-purity nitrogen gas.

A.4.4.2.8 Column front pressure: 0.06 MPa to 0.1 MPa.

A.4.4.2.9 Tail blowing flow rate: 20 mL/min.

A.4.4.2.10 Hydrogen flow rate: 30 mL/min.

A.4.4.2.11 Air flow rate: 300 mL/min.

A.4.4.3 Determination

Under the optimal conditions of the instrument, extract the sample solution and inject it into the gas chromatograph. Record the chromatogram and quantify it using the area normalization method.

A.4.5 Calculation of results

The mass fraction w of dibutyl hydroxytoluene content is calculated according to formula (A.1):

$$w = \frac{A}{\sum A_i} \times 100\% \quad \dots\dots\dots (A.1)$$

In which:

A - Peak area of dibutyl hydroxytoluene in the sample solution;

A_i - peak area of component i in the sample solution.

The measurement results are expressed as the arithmetic mean of two parallel measurements, and the absolute difference between the two parallel measurement results is not greater than 0.2%.

A.5 Determination of Free Phenols (calculated as p-cresol)

A.5.1 Reagents and materials

A.5.1.1 Bromine standard solution: $c(1/2Br_2) = 0.1$ mol/L.

A.5.1.2 Sodium thiosulfate standard titration solution: $c(Na_2S_2O_3) = 0.1$ mol/L.

A.5.1.3 Sodium hydroxide solution: 25 g/L.

A.5.1.4 Hydrochloric acid solution: 1+1.

A.5.1.5 Potassium iodide solution: 100g/L.

A.5.1.6 Starch indicator solution: 10g/L.

A.5.2 Instruments and equipment

Analytical balance: with a sensitivity of 0.0001g.

A.5.3 Analysis steps

Weigh about 10 g of the sample (accurate to 0.0001 g) into a 250 mL conical flask, add 50 mL of sodium hydroxide solution, and shake at 65 °C for no less than 3 minutes. Filter into a 250 mL volumetric flask and wash the filter residue with water several times; combine the filtrate in the volumetric flask and dilute with water to the mark. Take 100.00 mL into a 500 mL iodine flask, accurately add 10.00 mL of bromine standard solution, and add 10 mL of hydrochloric acid solution. Quickly cover and shake well, seal with 5 mL of potassium iodide solution, and set aside at 15 °C for 15 minutes. Put the potassium iodide solution into the iodine flask, cover and shake, and seal with water, set aside for 5 minutes. Titrate with sodium thiosulfate standard titration solution until light yellow, add 5 mL of starch indicator solution, and continue titrate until the blue color disappears as the endpoint. Simultaneously conduct a blank control experiment.

A.5.4 Calculation of results

The mass fraction w of free phenol (calculated as p-cresol) is calculated according to formula (A.2):

$$w = \frac{(V_2 - V_1) \times c \times 18.0}{m \times 1000 \times (100/250)} \times 100\% \dots\dots\dots(A.2)$$

In which:

V_2 : the volume of sodium thiosulfate standard titration solution consumed by blank solution, in milliliters (mL);

V_1 : the volume of sodium thiosulfate standard titration solution consumed by the sample, in milliliters (mL);

c : the concentration of standard titration solution of sodium thiosulfate, in mole per liter (mol/L);

m : mass of the sample, in grams (g);

18.0- Molar mass of cresol ($1/6 \text{ CH}_3\text{C}_6\text{H}_4\text{OH}$), in grams per mole (g/mol);

1000- Conversion factor;

100/250- Conversion factor.

The measurement results are expressed as the arithmetic mean of two parallel measurements, and the absolute difference between the two parallel measurement results is not greater than 0.002%.

A.6 Determination of Sulfate (calculated as SO_4^{2-})

A.6.1 Reagents and materials

A.6.1.1 Hydrochloric acid solution: 1+10.

A.6.1.2 Barium chloride solution: 50 g/L, prepare on site as required.

A.6.1.3 Sulfate (SO_4) standard solution: 0.1 mg/mL

A.6.2 Analysis steps

Weigh about 5 g of the sample (accurate to 0.01 g) into a 50 mL beaker, add 30 mL of freshly boiled and cooled water, and stir while heating until the sample dissolves; stir and cool to room temperature. Filter into a 50 mL colorimetric tube, add 5 mL of hydrochloric acid solution and 5 mL of barium chloride solution respectively, and dilute to 50 mL with freshly boiled and cooled water. Shake well and set aside for 10 minutes. Take another 1.00 mL of sulfate standard solution and treat it in the same way as the sample, using it as the standard solution. The turbidity of the sample solution shall not exceed the standard solution, which is $\leq 0.002\%$.

Appendix B

The Gas Chromatogram of the Standard Solution of Dibutyl Hydroxytoluene

B.1: Reference gas chromatogram of the standard solution of dibutyl hydroxytoluene

The reference gas chromatogram of the standard solution of dibutyl hydroxytoluene (50 mg/mL) is as shown in Figure B.1.

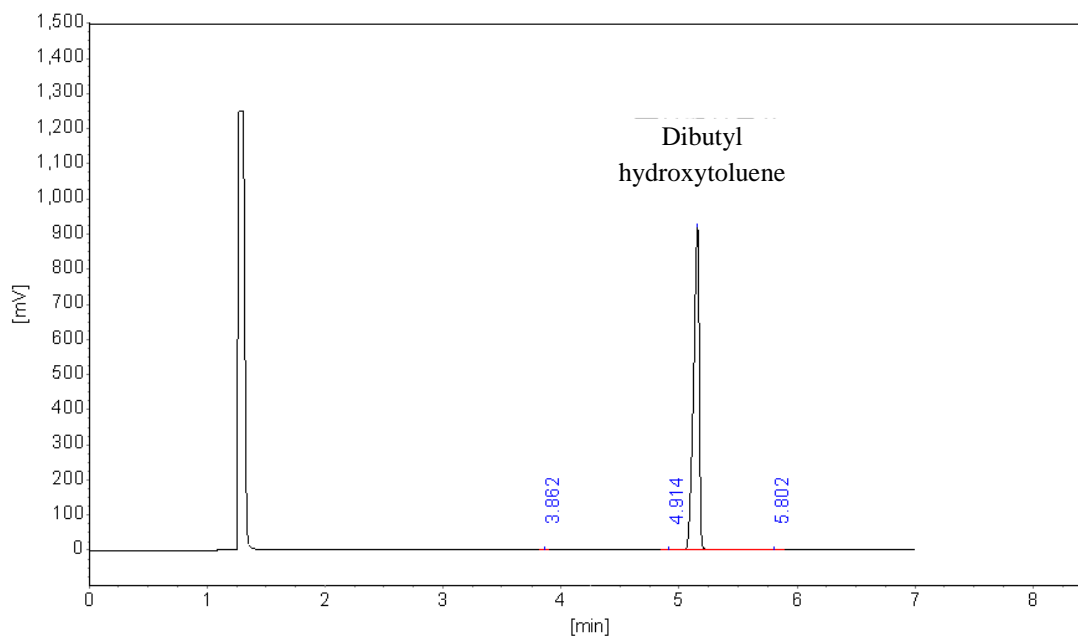


Figure B.1 Reference gas chromatogram of the standard solution of dibutyl hydroxytoluene (50 mg/mL)

END TRANSLATION

Attachments:

No Attachments.