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Date: 5/31/2011
GAIN Report Number:

China - Peoples Republic of

Post: Beijing

Food Additive Tertiary Butylhydroquinone (TBHQ)

Report Categories:
FAIRS Subject Report

Approved By:
Scott Sindelar

Prepared By:
Melinda Meador and Wu Bugang

Report Highlights:
On May 2, China's Ministry of Health notified to the WTO the National Food Safety Standard on Food Additive Tertiary Butylhydroquinonoe (TBHQ) as G/SPS/N/CHN/357. The standard specifies the technical requirements and testing methods for food additive TBHQ. It came into force on May 15, 2011. This report contains an INFORMAL translation of the document.

General Information:
BEGIN TRANSLATION

National food safety standard

Food Additive Tertiary Butylhydroquinone (TBHQ)

GB26403-2011

Issued on March 15, 2011

Implemented on May 15, 2011

Issued by the Ministry of Health

National Food Safety Standard

Food Additive

TBHQ

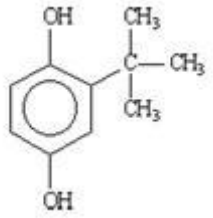
This Standard is applicable for Tert-butylhydroquinone (TBHQ for short), a kind of food additive using hydroquinone as raw material and produced through alkylation reaction.

2. Molecular Formula, Constitutional Formula and Relative Molecular Mass

2.1 Molecular Formula

C10H14O2

2.2 Constitutional Formula



2.3 Relative Molecular Mass

166.22 (as per 2007 International Relative Molecular Mass)

3. Technical Requirements

3.1 Sensory requirements shall conform to the requirements in Table 1.

Table 1 Sensory Requirements

Item	Requirements	Testing Method
Color	White	Put an appropriate amount of samples into a clean and dry white porcelain plate, and observe the color and status of samples in natural light, and then smell the samples.
Odor	Special Odor	
Status	Crystalline Powder	

3.2 Physicochemical indicators shall conform to the requirements in Table 2.

Table 2 Physicochemical Indicators

Item		Indicators	Testing Method
TBHQ (calculated as C10H14O2), *w*/%	≥	99.0	, Appendix A
T-butyl-benzoquinone, *w*/%	≤	0.2	, Appendix A
2,5－di-t-butyl-hydroquinone, *w*/%	≤	0.2	, Appendix A
Hydroquinone, *w*/%	≤	0.1	, Appendix A
Toluene/(mg/kg)	≤	25	, Appendi□ A
Lead (Pb)/(mg/kg)	≤	2	GB 5009.12
Fusion Point/℃		126.5～128.5	GB/T 617

Appendix A

Testing Method

A.1 General

Unless otherwise specified, any reagents and water involved in this Standard shall refer to analytical reagent and grade-III water as specified in GB/T 6682—2008. Unless otherwise specified, any standard volumetric solution, standard solution for impurity content test, preparation, and final products used for the test shall be prepared in accordance with the regulations under GB/T 601, GB/T 602 and GB/T 603. Any solution used for the test that the testers do not know which solvent will be used to prepare, shall refer to aqueous solution.

A.2 Identification Test

Dissolve 4mg~8mg samples into 1mL methanol, and then instill several drops of 25% dimethylamine aqueous solution. The solution turns from light red to red, and the color will not be returned.

A.3 Determination of TBHQ, T-butyl-benzoquinone, 2,5－di-t-butyl-hydroquinone, and Hydroquinone

A.3.1 Reagents and Materials

A.3.1.1 Acetone

A.3.1.2 Hydroquinone standard with known purity

A.3.1.3 TBHQ standard: Purity≥99%

A.3.1.4 T-butyl-benzoquinone standard: Purity≥99%

A.3.1.5 2,5－di-t-butyl-hydroquinone standard: Purity≥99%

A.3.2 Instruments and Equipment

Gas Chromatograph: Equipped with hydrogen flame ionization detector and automatic integrator.

A.3.3 Reference Chromatographic Conditions

A.3.3.1 Chromatographic Column: HP-5 elastic quartz capillary column, 30 m long, which inner diameter is 0.32 mm, and coating thickness is 0.25 μm; or other equivalent chromatographic columns.

A.3.3.2 Flow Rate: Carrier gas refers to high-purity nitrogen, which line speed is 30 cm/s.

A.3.3.3 Temperature: Column temperature is 220℃, sample inlet temperature is 250℃, and detector temperature is 300℃.

A.3.3.4 Splitting Ratio: 20:1.

A.3.3.5 Sample Size: 1 μL.

A.3.4 Analytical Procedure

A.3.4.1 Preparation of Standard Solutions

Take 10mg of hydroquinone, TBHQ, T-butyl-benzoquinone, and 2,5－di-t-butyl-hydroquinone standards respectively. Dissolve with acetone, and transfer to 10mL measuring flasks respectively. Dilute till the solution meets a certain scale, and shake up.

A.3.4.2 Preparing Sample Solution

Take 0.2g samples and dissolve with acetone. Transfer to a 10mL measuring flask. Dilute till the solution meets a certain scale, and then shake it up.

A.3.4.3 Determination

Under A3.3 reference chromatographic conditions, conduct meteorological and chromatographic analyses on all standard solutions, determine the retention time of all standards, and then feed 1μL sample solution for chromatographic analysis.

A.3.5 Calculation of Results

Calculate the contents of TBHQ, T-butyl-benzoquinone, 2,5－di-t-butyl-hydroquinone, and hydroquinone respectively with area normalization method.

The test result is subject to arithmetic mean of parallel determination results. Relative deviation of the determination result of TBHQ is no more than 0.2%, and relative deviation of determination results of other substances is no more than 2%.

A.4 Determination of Toluene

A.4.1 Reagents and Materials

A.4.1.1 Octanol.

A.4.1.2 Toluene.

A.4.2 Instruments and Equipment

Gas Chromatograph: Equipped with hydrogen flame ionization detector.

A.4.3 Reference Chromatographic Conditions

A.4.3.1 Chromatographic Column: Stainless steel packed column, 3.66m long, which outer diameter is 3.18 mm, and packing refers to SE-30-type silicone and diatomite-type insulation brick S (Diatoport S) (180μm~250μm), by weight, ratio is 10:100; or other equivalent chromatographic columns.

A.4.3.2 Carrier gas refers to nitrogen, which flow rate is 25 mL/min.

A.4.3.3 Temperature: Column temperature is 70℃, sample inlet temperature is 275℃, and detector temperature is 300℃.

A.4.3.4 Splitting Ratio: 50:1.

A.4.3.5 Sample Size: 1 μL.

A.4.4 Analytical Procedure

A.4.4.1 Preparation of Sample Solution

Take about 0.2g samples, accurate to 0.0002g. Transfer to a 10mL measuring flask and dissolve with octanol. Dilute till the solution meets a certain scale, and then shake it up. Take cS as accurate concentration.

A.4.4.2 Preparation of Standard Solution

Prepare octanol solution containing 50μg toluene per mL. Take cR as accurate concentration.

A.4.4.3 Determination

Under A4.3 reference chromatographic conditions, conduct chromatographic analysis on standard solution and sample solution respectively. Measure the peak height of toluene (*HR*) on chromatogram of standard solution, and other peaks are irrelevant to the analysis. Similarly, measure the peak height of toluene (*HS*) on chromatogram of sample solution.

A.4.5 Result Calculation

Toluene content is recorded as *w*—mass fraction of toluene in mg/kg, and calculated with Formula (A.1):

$$w = (H_S / H_R) \times (c_R / c_S) \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots \quad (A.1)$$

Where,

HS-Numerical value of toluene peak height on sample solution chromatogram;

HR-Numerical value of toluene peak height on standard solution chromatogram;

cR-Numerical value of toluene concentration in standard solution, μg/mL;

cS-Numerical value of sample solution concentration, g/mL;

The test result is subject to arithmetic mean of parallel determination results. Relative deviation of parallel determination results is no more than 10%.

END TRANSLATION