



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

GB 1886.59-2015

**National Food Safety Standard -- Food Additives -- Petroleum
ether**

食品安全国家标准 食品添加剂 石油醚

(English Translation)

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1 Scope

This standard applies to natural petroleum fractions prepared food additive petroleum ether. The product is at a boiling point between 25 °C ~ 105 °C of Light petroleum products, mainly for mixed alkanes (n homogeneous and heterogeneous) and naphthenes.

2.1 Sensory requirements

Sensory requirements shall comply with the requirements of Table 1.

Table 1 Sensory requirements

Project requires test methods Color colorless Transparent liquid state Odor similar to the smell of gasoline Take appropriate sample in a clean, dry colorimetric tube, in natural light by visually observe its color and status, olfactory odor

2.2 Physical indicators

Physical and chemical indicators should be consistent with the provisions of Table 2.

Table 2. Physical and chemical indicators

Item Index Test Method Evaporation residue/(mg/100mL) ≤ 2 GB/T 6324.2 S/(mg/kg) ≤ 10 GB/T 6324.4 Benzene, phi /% ≤ 0.05 Appendix A A.4 Aromatic hydrocarbons (benzene), phi /% ≤ 0.3 Appendix A A.4 Bromine index/(mg/100g) ≤ 200 Appendix A A.5 Lead (Pb)/(mg/kg) ≤ 2.0 GB 5009.12 PAHs test by GB 7363 Appendix A Testing method A.1 Warning Some test procedures prescribed test method can lead to dangerous situations. The operator shall take appropriate safety and health practices.

A.2 General Provisions Unless otherwise indicated in the analysis using only recognized as analytical reagents and GB/T 6682 stipulated three water. Test Methods the standard titration solution, impurity measurement standard solution, preparations and products, did not indicate when the other requirements, according to GB/T 601, GB/T 602 Preparation and provisions GB/T 603; and the solution, unless otherwise indicated, are the solution.

A.3 Identification Test Insoluble in water, soluble in ethanol.

A.4 benzene and aromatics (benzene) Determination A.4.1 Method summary by gas chromatography under the selected chromatographic conditions, sample after vaporization separated by capillary column with flame ionization detector Detection with internal standard.

A.4.2 Reagents and materials A.4.2.1 Hydrogen. the volume fraction of not less than 99.99%, silica gel and molecular sieves, purified.

A.4.2.2 nitrogen. the volume fraction of not less than 99.99%, silica gel and molecular sieves, purified.

A.4.2.3 Air. silica gel and molecular sieves, purified.

A.4.2.4 isooctane. $\geq 99\%$, aromatic substance containing less than 0.05%.

A.4.2.5 internal standard. n- decane and n- undecane, chromatography; or n- decane and n- dodecane, chromatography.

A.4.2.6 standard solution A. 0.5% of the internal standard solution and 0.5% iso-octane benzene.

A.4.2.7 Standard solution B. 0.5% of n- decane, 0.5% of the internal standard solution and 0.5% iso-octane benzene.

A.4.3 Instruments and Equipment A.4.3.1 Gas chromatograph. with a capillary column injector device and a flame ionization detector.

A.4.3.2 micro syringe. 10 μ L.

A.4.4 columns and operating conditions the recommended standard chromatographic columns and the operating conditions are shown in Table A.1, n- decane can before the same degree of separation of the benzene peak column and chromatographic operating conditions can also be used.

Table A.1 Recommended chromatographic columns and operating conditions

Project Parameters

Column pentaerythritol -beta- cyanide ether capillary column Column length 30m x 0.25mm x column diameter Column temperature/ $^{\circ}$ C 95 Vaporization chamber temperature/ $^{\circ}$ C 275 Detector temperature/ $^{\circ}$ C 250 Carrier gas (nitrogen) flow rate/(mL/min) 3 Split ratio 100.1 Injection volume/ μ L 10 Determination of the internal standard response factor with respect to benzene A.4.5 Benzene with respect to the internal standard response factor to f expressed by the formula (A.1) Calculated.

A1 x w

w1 x A

Where.

A1 --- internal standard peak area;

w --- A standard solution mass fraction of benzene;

W1 --- quality within the standard solution A standard material fraction;

A --- benzene peak area.

A.4.6 Analysis step

Approximately 0.1mL internal standard into a 25mL volumetric flask, using analytical balance. With the test sample was dissolved and diluted to the mark. press

Table A.1 given operating conditions inject 10μL sample containing internal standard. Before measuring the internal standard peak area and benzene, change the instrument failure

Save to ensure that the peak of the peak of at least 25% of full scale. Measurement and benzene internal standard peak area. Paul and typical chromatograms of the components the residence time in Appendix B in Figure B.1 and Table B.1.

A.4.7 Quantitative Analysis Using internal standard.

A.4.8 Calculation Results the mass fraction of a measured component w_i , according to equation (A.2) Calculated.

$A_i \times f \times m_1$

$A_2 \times m \times$

100% (A.2)

Where.

A_i --- a peak area of component being tested;

f --- benzene with respect to the response factor of the internal standard;

m_1 --- added mass of the internal standard in grams (g);

A_2 --- internal standard peak area;

M --- the quality of the sample, in grams (g).

A.5 Determination of bromine index A.5.1 Method summary Aware of the large number of sample was dissolved by the standard bromide - bromate special solution titrate in. When there is the presence of free chlorine, causing the system A sudden change in conductivity, potentiometric titrator indicate the end. Bromine index refers 100g sample occurred under the test conditions of the reaction mass of bromine , And the unit of mg/100g.

A.5.2 Reagents and materials A.5.2.1 mixed solvent. A 714mL glacial acetic acid, 134mL of carbon tetrachloride, 134mL of methanol and 18mL of sulfuric acid (15) were mixed.

A.5.2.2 sodium thiosulfate standard titration solution. $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.05\text{mol/L}$.

A.5.2.3 bromide - potassium bromate standard solution. $c(6\text{KBr-KBrO}_3) = 0.05\text{mol/L}$. Weigh 5.1g 1.4g potassium bromide and potassium bromate, Diluted with water to 1000mL. 1mL to 50mL glacial acetic acid and hydrochloric acid test solution was added to 500mL iodine bottle. Cooled in an ice-water bath Possession of about 10min, and spinning iodine bottle, add 40mL ~ 50mL (accurate to 0.01mL) of bromide from 50mL burette -Bromate solution, speed control between 90s ~ 120s. Quickly covered with caps, mix a solution, and this placed in an ice water bath. In the iodine bottle the bottle plus 5.0mL15% potassium iodide solution. 5min after iodine bottle was removed from the ice water bath and remove the stopper, so that 15% of the iodide Potassium iodine solution is slowly flowing into the bottle. Mix well with clean water 100mL cork, bottle and the bottle wall, and immediately with sulfur 0.05mol/L of Sodium thiosulfate standard solution titration, near the end added starch test solution slowly titrated to a blue color disappears, is the end.

Potassium bromide - potassium bromate standard solution concentration c_1 , according to formula (A.3) Calculated.

$V_2 \times c_2$

Where.

V_2 --- consumption volume of sodium thiosulfate standard titration solution, in milliliters (mL);

Concentration c_2 --- sodium thiosulfate standard titration solution, expressed in moles per liter (mol/L);

V_1 --- added potassium bromide - potassium bromate standard solution volume, in milliliters (mL).

A.5.3 Instruments and Equipment A.5.3.1 titrator.

A.5.3.2 titration vessel. capacity of about 150mL, and can be maintained with a glass casing $1^\circ\text{C} \sim 5^\circ\text{C}$ condensation plant. Two platinum electrodes Place the distance is not greater than 5mm, and depth below the liquid level. Mechanical or magnetic stirrer, and stirred sufficiently not cause the bubbles.

A.5.3.3 dropper. capacity of 10mL and 50mL.

A.5.3.4 iodine bottle. with a glass stopper, capacity 500mL.

A.5.4 Analysis step

With a suitable cooling fluid through the casing titration vessel, circulating cooling titration vessel maintained at $1^\circ\text{C} \sim 5^\circ\text{C}$. The mixture was added 110mL Co-solvent (A.5.2.1) and a sample of 8g ~ 10g. Open stirrer, stirring rate adjusted to the solution, but not from the whirlpool bubbles appropriate.