



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

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National food safety standard - Food additives - Phospholipid

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1 Scope

This document is applicable to phospholipid, a food additive, that uses soybean, sunflower seeds, rapeseed and other vegetable oilseeds or their processing by-products as the main raw materials, that is obtained by dehydration, decontamination, decolorization or degreasing, or using egg yolk as the main raw material through extraction, refining and other processes.

2.1 Sensory requirements

Sensory requirements should meet the requirements of Table 1.

2.2 Physical and chemical indicators

Physical and chemical indicators should meet the requirements of Table 2.

Annex A Inspection methods

A.1 General The reagents and water used in this Standard, unless other requirements are specified, all refer to analytically pure reagents and grade three water specified in GB/T 6682-2008. The standard titration solution, standard solution for impurity determination, preparations and products used in the test, unless other requirements are specified, should be prepared in accordance with the provisions of GB/T 601, GB/T 602 and GB/T 603.

A.2 Identification test A.2.1 Take 2mL of ethanol solution containing 0.5% sample into a test tube. Add 1~2 drops of 5% cadmium chloride ethanol solution to produce a white precipitate.

A.2.2 Take 2mL of ethanol solution containing 10% sample into a test tube. Add 1~2 drops of potassium bismuth nitrate solution (Take 8g of bismuth nitrate. Add 20mL of nitric acid to dissolve. Take 27.2g of potassium iodide. Add 50mL of water to dissolve.

Combine the above two solutions. Add water to dilute to 100mL) to produce a brick red precipitate.

A.3 Determination of n-hexane insoluble matter A.3.1 Reagents and materials N-hexane.

A.3.2 Instruments and equipment A.3.2.1 Suction filter. 500mL.

A.3.2.2 Glass sand core crucible (funnel). G3.

A.3.3 Analysis steps A.3.3.1 Bake the cleaned crucible to constant weight in a 101°C ~ 105°C oven.

A.3.3.2 Weigh 10.0g of specimen, to the nearest of 0.0001g. Place in a beaker. Add about 100mL of n-hexane. Use a glass rod to stir and dissolve. Conduct suction-filtration through a constant weight crucible.

A.3.3.3 Use 25mL of n-hexane to wash the beaker and glass rod twice. Transfer all the insoluble matter into the crucible. Use n-hexane to wash the inner wall of the crucible and insoluble matter. Finally, try to extract the residual n-hexane in the crucible.

A.3.3.4 Remove the crucible. Use absorbent cotton dipped in a little n-hexane to wipe the outer wall of the crucible. Bake the crucible in an oven at 101°C ~ 105°C for 1h.

Take out. Put in a desiccator and cool to room temperature. Weigh. Dry for another 20min. Cool. Weigh till constant weight.

A.3.4 Result calculation The content of n-hexane insoluble matter is calculated in mass fraction w_1 and calculated according to formula (A.1).

Where, m_1 - Total mass of empty crucible and insoluble matter, in grams (g);

m_2 - Mass of empty crucible, in grams (g);

m - mass of specimen, in grams (g).

The test result is based on the arithmetic mean of the parallel measurement results. The calculation result has two significant figures. The absolute difference between the results of two independent determinations obtained under repeatability conditions should not exceed 5% of the arithmetic mean.

A.4 Determination of acid value A.4.1 Reagents and materials A.4.1.1 Petroleum ether.

A.4.1.2 Standard titration solution of sodium hydroxide. $c(\text{NaOH}) = 0.1\text{mol/L}$.

A.4.1.3 Neutral ethanol. the mass fraction is 95%. Use phenolphthalein indicator solution as indicator before use. Use sodium hydroxide standard titration solution to titrate until the solution turns reddish. Maintain 5s without fading as the end point.

A.4.1.4 Phenolphthalein indicator solution. 10g/L.

A.4.2 Analysis steps Weigh about 2g of the well-mixed specimen, to the nearest of 0.001g. Place in a 250mL conical flask. Add 50mL of petroleum ether. Shake gently to dissolve. Then add 50mL of neutral ethanol. Shake well. Add 4 drops of phenolphthalein indicator

solution. Use sodium hydroxide standard titration solution to quickly titrate. Shake while adding drops. When approaching the end point, slow down the titration speed to 1~2 drops each time. The end point is when the pink color appears and does not fade for 5s.

solution to 30mL of acetic acid and chloroform mixture. If the solution appears blue and requires more than 1 drop of sodium thiosulfate standard titration solution to eliminate, the solution needs to be reconstituted.

A.5.1.3 Standard titration solution of sodium thiosulfate. $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.01 \text{ mol/L}$. It is prepared by diluting the standard titration solution of sodium thiosulfate with $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.1 \text{ mol/L}$ by 10 times.

A.5.1.4 Starch indicator solution. 10g/L.

NOTE. All the above reagents and water must not contain dissolved oxygen.

A.5.2 Instruments and equipment Laboratory routine instruments. All utensils used must not contain reducing or oxidizing substances. The frosted glass surface must not be oiled.

A.5.3 Analysis steps Weigh about 5g of the evenly mixed specimen, to the nearest of 0.001g. Place in a 250mL iodine bottle. Add 30mL of acetic acid and chloroform mixture. Shake to fully dissolve the specimen. Add 0.5mL of saturated potassium iodide solution. Tightly cap and react for 1min. Shake the iodine bottle gently at least 3 times during the reaction.

Then immediately add 30mL of water and 0.5mL of starch indicator solution. Use sodium thiosulfate standard titration solution to titrate. Shake while adding drops.

Towards the end point, shake continuously to release all the iodine from the solvent layer. Add the sodium thiosulfate standard titration solution dropwise until the blue color of the solution disappears, which is the end point. Conduct blank test at the same time. When the standard titration solution of sodium thiosulfate consumed by the blank test exceeds 0.1mL, the reagent should be replaced, and the specimen should be measured again.

A.5.4 Result calculation The peroxide value is calculated in w_3 , and the value is expressed in millimoles per kilogram (mmol/kg) and is calculated according to formula (A.3).

Where, 1000 - Conversion factor;

V - Volume of the standard titration solution of sodium thiosulfate consumed by the titration specimen, in milliliters (mL);