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NATIONAL STANDARD OF THE
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GB 28401-2012

National Food Safety Standard Food Additives - Phospholipids

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Appendix A

Test Methods

A.1 General

Unless otherwise specified, reagent and water in this Standard refer to analytic reagent and Grade-3 water as specified in GB/T 6682-2008. Unless otherwise specified, standard titration and reagent solutions used for determination of impurities shall be prepared in accordance with GB/T601, GB/T 602 and GB/T 603. The solution used in test which is not indicated with preparation method refers to aqueous solution.

A.2 Identification test

A.2.1 Put 2ml of ethanol solution (0.5%) in test tube; add 1 to 2 drops of cadmium chloride. White precipitate is generated.

A.2.2 Put 2ml of ethanol solution (0.5%) in test tube; add 1 to 2 drops of bismuth potassium nitrate solution (take 8g of bismuth nitrate; add 20ml of nitric acid to dissolve; take 27.2 g of potassium iodide; add 50ml of water to dissolve; Mix the said two solutions; add water to 100 ml). Brick-red precipitate is generated.

A.3 Test of insoluble substance — n-hexane

A.3.1 Reagent and substance

n-hexane.

A.3.2 Instruments

A.3.2.1 Suction filter: 500 ml.

A.3.2.2 Sand core glass crucible: G3.

A.3.3 Operation steps

A.3.3.1 Put a clean crucible in a 101°C ~ 105°C drying oven to bake to constant weight.

A.3.3.2 Take 10.0g of mixed samples (accuracy: 0.0001g); put it in beaker. Add 100ml of n-hexane; stir the solution with glass rod to fully dissolve. Extract and filter through constant-weight crucible.

A.3.3.3 Use total 25ml of n-hexane to clean beaker and glass rod for 2 times; transfer the insoluble substances into crucible. Use n-hexane to wash the internal wall of crucible and insoluble substances. Finally remove the residual n-hexane left inside crucible as

reductive or oxidizing substances. The surface of ground glass shall not be coated with oil.

A.5.3 Operation steps

Take 5g of mixed samples (accurate to 0.001g); put it in a 250 mL iodine flask. Add 30ml of mixed solution of glacial acetic acid and chloroform. Shake it to fully dissolve. Add 0.5ml of saturated potassium iodide solution; put cover on it. Wait for 1min for reaction. At least shake 3 times during the reaction process. Add 30ml of water and 0.5ml of starch indicator. Use sodium thiosulfate standard titration solution to titrate the solution; keep shaking during titration. When it is closing to end, keep shaking to make all of iodine released from solvent layer. Add sodium thiosulfate standard titration solution drop-by-drop, until the solution's blue color fades away as the end. At the same time, conduct blank test. When the consumed sodium thiosulfate standard titration solution exceeds 0.1ml for blank test, it shall replace reagent and re-conduct the test.

A.5.4 Calculation

Peroxide value w_3 , the value is expressed by milligram equivalent of active oxygen per kilogram meq/kg, is calculated according to Formula (A.3):

$$w_3 = \frac{1000 \times (V - V_0) \times c}{m_3} \dots\dots\dots (A.3)$$

Where:

V — the volume of consumed sodium thiosulfate standard titration solution, unit: ml;

V_0 — the volume of consumed sodium thiosulfate standard titration solution used in blank test, unit: ml;

c — the accurate concentration of sodium thiosulfate standard titration solution, unit: mol/L;

m_3 — the mass of sample, unit: g;

1000 — conversion factor.

The arithmetic mean of the parallel determination results is the test result. The absolute difference of two independent test results must not exceed 5% of the calculated arithmetic mean, under repeatability conditions.

END

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