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

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Replacing GB/T 5539-2008

Inspection of grain and oils - Qualitative test of oils

粮油检验 油脂定性试验

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1 Scope

This document describes qualitative methods of detection for vegetable oils and fats.

It applies to the detection of tung oil, castor oil, linseed oil, mineral oil, sesame oil and cottonseed oil.

2 Normative references

The contents of the following documents constitute indispensable provisions of this document through normative reference in the text. For dated references, only the edition corresponding to that date applies. For undated references, the latest edition, including all amendments, applies.

GB/T 6682 Water for analytical laboratory use - Specification and test methods · GB/T 22515 Grain and oils terminology - Grain, oilseeds and their products

3 Terms and definitions

The terms and definitions given in GB/T 22515, together with the following, apply to this document.

mineral oil: base oil refined from petroleum by physical distillation.

4.1 Detection of tung oil

Reagent: concentrated sulfuric acid, analytical grade.

Apparatus: white spot plate, with six, nine or twelve wells.

Procedure: place a few drops of the well-mixed sample in a well of the white spot plate, add one or two drops of concentrated sulfuric acid and observe the change of colour.

Interpretation: a deep red solid whose colour deepens gradually and turns black after 15 min to 20 min shows that tung oil is present; the deeper the colour, the higher its content. The method is suitable for detecting tung oil at contents above 0.5 %.

4.2 Detection of castor oil

Reagents: potassium hydroxide and magnesium chloride, both analytical grade.

Apparatus: flat-bottomed nickel crucible 4 cm in diameter and 3.5 cm high, and a balance readable to 0.01 g.

Procedure: weigh about 1.00 g of the well-mixed sample into the nickel crucible, add 0.03 g to 0.10 g of potassium hydroxide, heat slowly until it melts and smell it. Alternatively, dissolve the melt in water, add an excess of magnesium chloride to precipitate the fatty acids, filter and observe whether the filtrate is turbid.

Interpretation: a smell of octanol in the first procedure, or a turbid filtrate in the second, shows that castor oil is present. The method is suitable for detecting castor oil at contents above 0.5 %; it does not apply to the detection of castor oil in peanut oil.

4.3 Detection of linseed oil

Reagents: diethyl ether, analytical grade; bromine solution, made by adding enough bromine to carbon tetrachloride to increase the volume of the solution by half, that is to 1.5 times the original volume.

Apparatus: 20 mL stoppered colorimetric tube, 100 microlitre to 1 000 microlitre micropipette and 10 mL measuring cylinder.

Procedure: pipette 0.5 mL of the well-mixed sample into the 20 mL stoppered colorimetric tube, add 10 mL of diethyl ether and 3 mL of bromine solution, stopper once dissolved, mix by inversion, bring the solution to 25 degrees Celsius and observe for 2 min. At the same time run a control on a normal sample free of linseed oil.

Interpretation: turbidity within 2 min shows that linseed oil is present. The method is suitable for detecting linseed oil at contents above 0.5 %; it does not apply to the detection of linseed oil in walnut oil, olive oil or sesame oil.

4.4 Detection of mineral oil

Reagents: water to grade three of GB/T 6682; absolute ethanol and potassium hydroxide,

both analytical grade; aqueous potassium hydroxide solution made by adding 15 g of potassium hydroxide, weighed to 0.01 g, to 10 mL of water.

Apparatus: water bath covering room temperature to 99.9 degrees Celsius, straight condenser, 250 mL ground-joint conical flask matching the condenser, 10 mL and 25 mL measuring cylinders, 1 mL micropipette and a balance readable to 0.01 g.

Procedure: weigh 1.00 g of the well-mixed sample to 0.01 g into the ground-joint conical flask, add 1 mL of the potassium hydroxide solution, 25 mL of absolute ethanol and two to four boiling stones, connect the reflux condenser and reflux in a water bath at about 97 degrees Celsius for about 5 min, shaking continuously until saponification is complete. Then add 25 mL of boiling distilled water to the flask at once, shake and observe immediately.

Interpretation: marked turbidity, or oily matter separating out, shows that mineral oil is present in the sample. The method is suitable for detecting mineral oil in vegetable oils and fats whose wax ester content is below 0.5 %, with a lower detection limit of 0.5 %. It does not apply to the detection of mineral oil in crude rice bran oil or in sea buckthorn seed oil.

4.5 Detection of sesame oil

Reagents: concentrated hydrochloric acid, analytical grade; 2 % furfural in ethanol, made by adding 2 mL of furfural to 95 % ethanol and making up to 100 mL.

Apparatus: 25 mL colorimetric tube, 10 mL and 25 mL measuring cylinders, 20 microlitre to 200 microlitre micropipette and 100 mL volumetric flask.

Procedure: measure 5 mL each of the well-mixed sample and of concentrated hydrochloric acid into the colorimetric tube, add 0.1 mL of the 2 % furfural solution, shake for 30 s to mix thoroughly, let stand for 10 min and observe the colour; where a deep red, red or pink colour appears, add 10 mL of water, shake again for 30 s and observe the colour. Dark oil samples may be bleached with alkali, the alkali and the water then being removed completely from the oil; where necessary a bleached sample containing sesame oil may be used as a control.

Interpretation: if the deep red, red or pink colour disappears, no sesame oil is present; if it does not disappear, sesame oil is present. The method is suitable for detecting sesame oil at contents above 0.25 %. It does not apply to the detection of sesame oil in olive oil.

4.6 Detection of cottonseed oil

Reagents: water to grade three of GB/T 6682; 1 % sulfur powder in carbon disulfide, made by mixing 1 g of sulfur powder into 100 mL of carbon disulfide; pyridine or amyl alcohol, analytical grade; and saturated brine.

Apparatus: 25 mm by 150 mm test tube, thermostatic water bath and 10 mL and 25 mL measuring cylinders.

Procedure: measure 5 mL each of the well-mixed sample and of the 1 % sulfur powder solution into the test tube, add two drops of pyridine or amyl alcohol, shake to mix, place in a bath of saturated brine and heat slowly until the brine begins to boil; after 40 min take the tube out and observe the change of colour.

Interpretation: a deep red or orange-red colour shows that cottonseed oil is present; the deeper the colour, the higher its content. The method is suitable for detecting cottonseed oil at contents above 0.5 %. It does not apply to the detection of cottonseed oil in dark oils such as sesame oil and crude corn oil.