Regional Economic Cooperation and Integration in Asia

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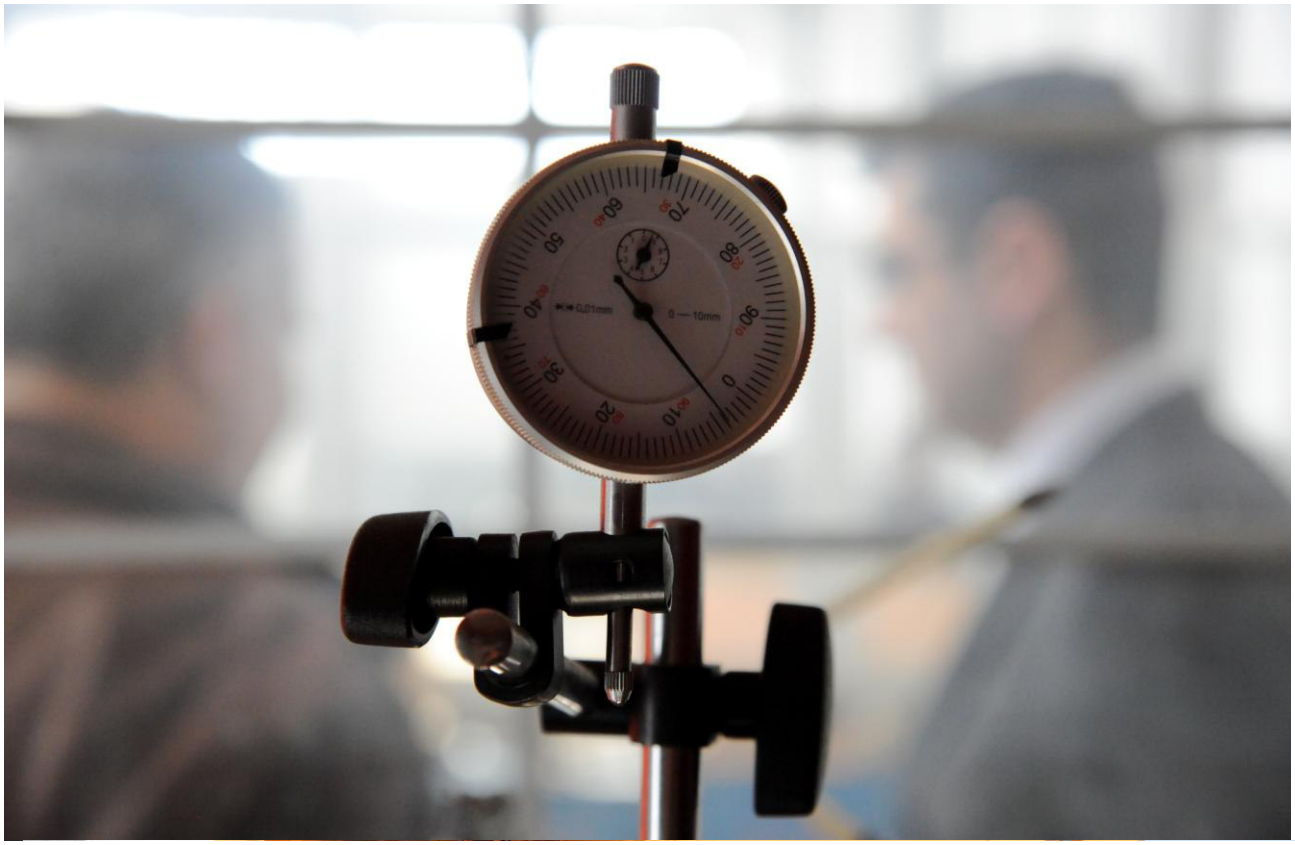

National Standard of People's Republic of China

GB 2715-2005

Hygienic standard for grains

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On behalf of the German Federal Ministry for Economic Cooperation and Development (BMZ)

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National Standard of the People's Republic of China

GB 2715 – 2005

Replaces GB 2715 – 1981

Hygienic standard for grains

Issue date: January 25, 2005 Implementation date: October 1, 2005

Issued by the Ministry of the Health of the People's Republic of China

Foreword

This standard is mandatory in full text.

This standard will replace GB 2715 -- 1981: Hygienic standard for grains, which was revoked.

The main changes in this standard compared with GB 2715 -- 1981 are as follows:

- Adding the scope of this standard as “This standard is applicable to raw grain and processed grain for human consumption including cereal, Legumes and tuber crops. This standard is not applicable to raw materials for edible oil processing;
- Adding the hygiene requirements of packaging, marking, transportation and storage;
- Adding the indexes of heat-damaged kernels and moldy kernels;
- Adding the index limits of ergot, lolium temulentum and stramonium seeds and other seeds of poison plant;
- Adding the limits of deoxynivalenol, zearalenone and ochratoxin A;
- Adding the maximum residue limits of methyl bromide, malathion, chlorpyrifos-methyl, pirimiphos-methyl, deltamethrin, lindane. Changing the indexes of total arsenic into inorganic arsenic. Deleting the indexes of cyanide and carbon disulfide.

This standard shall be effective on 1st October 2005; Transition period will be one year. Those products manufactured before 1st October 2005 but meeting the requirements of relevant standards are allowed to sell until 30th September, 2006.

Annex A of this standard is normative.

This standard is proposed and administered by the Ministry of Health of the People’s Republic of China.

The main organizations that participated in the drafting of this standard are Jiangsu Provincial Center for Disease Prevention and Control, National Center for Health Inspection and Supervision, Standards &Quality Center of the State Administration of Grain, National Institute of Nutrition and Food Safety of Chinese Center for Disease Prevention and Control, Grain Monitoring Center of the Ministry of Agriculture,

Liaoning Inspection and Quarantine Bureau, or Liaoning Entry-Exit Inspection and Quarantine Bureau of People's Republic of China in full (LNCIQ), Shanghai Entry-Exit Inspection and Quarantine Bureau.

Main drafters of this standard are Baojun Yuan, Yunyan Zheng, Huamin Xie, Xiahui Li, Tianliang Hou, Yuliang Guan, Ying Zhang, and Xuqing Wang.

The previous edition(s) replaced by this standard is (are) as follows:

- GB n1-1977
- GB 2715-1981

Hygienic Standard for Grains

1. Scope

This standard defines the hygiene indexes of grains, testing methods and hygiene requirements of packaging, marking, transportation and storage.

This standard is applicable to raw grains and processed grains for human consumption including cereals, legumes and tuber crops. This standard is not applicable to raw materials for edible oil processing.

2. Normative References

The terms of the following documents are by reference as the provisions of this standard. All subsequent modification lists (excluding corrigendum content) or revised editions of reference documents with date notes are not applicable to this standard. However, all parties entering into an agreement based on this standard are suggested to study whether the latest editions of these reference documents are applicable. The latest editions of all reference documents without date notes are applicable to this standard.

- GB 2760 Hygienic standard for uses of food additives
- GB 2763 Maximum residue limits for pesticides in Food
- GB/T 5009.11 Determination of total arsenic and abio-arsenic in foods
- GB/T 5009.12 Determination of lead in foods
- GB/T 5009.15 Determination of cadmium in foods
- GB/T 5009.17 Determination of total mercury and organic mercury in foods
- GB/T 5009.19 Determination of HCH and DDT residues in foods
- GB/T 5009.20 Method for determination of organophosphorus pesticide residues in foods
- GB/T 5009.22 Method for determination of aflatoxin B1 in foods
- GB/T 5009.36 Method for analysis of hygienic standard of grains
- GB/T 5009.96 Determination of ochratoxin A in cereals and soybeans
- GB/T 5009.110 Determination of cypermethrin,fenvalerate and

deltamethrin residues in vegetable foods

- GB/T 5009.111 Determination of deoxynivalenol in cereal and cereal products
- GB/T 5009.145 Determination of organophosphorus and carbamate pesticide multiresidues in vegetable foods
- GB/T 5494 Inspection of grain and oilseeds; Methods for determination of foreign matter and unsound kernels
- GB 7718 General standard for the labeling of prepackaged foods
- GB 13122 Hygienic specifications of flour milling factory
- SN 0649 Method for the determination of methyl bromide residues in cereals for export
- SN/T 0080.7 Cereals and feedstuffs for import and export--Method for the inspection of imperfect grains

3. Terms and definitions

The following terms and definitions are applicable to this standard.

3.1 Heat damaged kernel

Kernel whose color is changed into an abnormal one due to heat produced by microorganism or other reasons

3.2 Ergot

Sclerotium grown from fungi parasitizing in the ovary of gramineous plants

3.3 Lolium temulentum

Caryopsis of gramineae herb

3.4 Moldy kernel

Kernel with no edible value whose surface is clearly moldy and embryo and endosperm (or cotyledon) are damaged

4.Requirements on indexes

4.1 Sensory requirements

The color, smell and hygiene requirements for grains should reach the ordinary levels

and meet the requirements in Table 1.

Table 1 Sensory requirement for grains

Item		Index
Heat damaged kernel/(%)		
Wheat	≤	0.5
Moldy kernel/(%)	≤	2.0

4.2 Indexes of poisoned and harmful fungus and plant seeds

It should meet the requirements in Table 2.

Table 2 Indexes of poisoned and harmful fungus and plant seeds

Item		Index
Ergot/(%)		
Rice, corns and beans	≤	Not allowed to be detected
Wheat, Barley	≤	0.01
Darnel/(seed/kg)		
Wheat and barley	≤	1
Mandala seeds and other seeds of poisoned plants (seed/kg)		
Beans	≤	1

4.3 Physicochemical indexes

4.3.1 Index of fungaltoxin limit

It should meet the requirements in Table 3.

Table 3 Index of fungaltoxin limit

Item		Limit/(μg/kg)
Aflatoxin B_1		
Corn	≤	20

Rice	≤	10
Other	≤	5
Deoxynivalenol (DON) Wheat, barley, corn and processed grains	≤	1000
Zearalenone Wheat and corns	≤	60
Ochratoxin A Cereals and beans	≤	5

4.3.2 Index of pollutant limit

It should meet the requirements in Table 4.

Table 4 Index of pollutant limit

Item		Limit/(mg/kg)
Lead (Pb)	≤	0.2
Cadmium (Cd) Paddy (including rice) and beans Wheat (including wheatmeal), corns and others	≤ ≤	0.2 0.1
Mercury (Hg)	≤	0.02
Inorganic arsenic (As) Rice Wheatmeal Others	≤ ≤ ≤	0.15 0.1 0.2

4.3.3 Maximum residue limit of pesticide

It should meet the requirements in Table 5.

Table 5 Maximum residue limit of pesticide

Item		Maximum residue limit /(mg/kg)
Phosphide (PH_3)	≤	0.05
Methyl bromide	≤	5
Malathion Rice	≤	0.1
Chlorpyrifos-methyl	≤	5
Pirimiphos-methyl Wheat and grains	≤	5
Deltamethrin	≤	0.5
Benzex	≤	0.05
Lindane Wheat	≤	0.05
DDT	≤	0.05
Nitrochloromethane (raw grains)	≤	2
Heptachlor	≤	0.02
Aldrin	≤	0.02
Dieldrin	≤	0.02
Other pesticides		See the requirements of GB 2763

5. Food additives

5.1 The quality of food additives should meet the requirements of relevant standards or certain regulations.

5.2 The kinds and usage amount of food additives should meet the requirements of GB 2760.

6 Testing methods

6.1 Sensory testing

Follow the testing method in GB/T 5009.36.

6.2 Heat damaged kernel

Follow the testing method in SN/T 0080.7.

6.3 Moldy kernel

Follow the testing method in GB/T 5494.

6.4 Ergot, darnel, mandala seed and other seeds of poisoned plants

Follow the testing method in GB/T 5009.36.

6.5 Aflatoxin B1

Follow the testing method in GB/T 5009.22.

6.6 Deoxynivalenol

Follow the testing method in GB/T 5009.111.

6.7 Zearalenone

Follow the testing method in Annex A.

6.8 Ochratoxin A

Follow the testing method in GB/T 5009.96.

6.9 Inorganic arsenic

Follow the testing method in GB/T 5009.11.

6.10 Lead

Follow the testing method in GB/T 5009.12.

6.11 Cadmium

Follow the testing method in GB/T 5009.15.

6.12 Mercury

Follow the testing method in GB/T 5009.17.

6.13 Phosphide, heptachlor, aldrin, dieldrin and nitrochloroform

Follow the testing method in GB/T 5009.36.

6.14 Methyl bromide

Follow the testing method in SN 0649.

6.15 Malathion

Follow the testing method in GB/T 5009.110.

6.16 Chlorpyrifos-methyl and pirimiphos-methyl

Follow the testing method in GB/T 5009.145.

6.17 Deltamethrin

Follow the testing method in GB/T 5009.110.

6.18 Benzex, DDT and Lindane

Follow the testing method in GB/T 5009.19.

7 Processing procedure of processed grains

Follow the testing method in GB 13122.

8 Packaging

Packaging of grains shall use materials or containers which meet certain hygiene requirements. Packaging should be intact, undamaged and unpolluted.

9. Labelling

Labels of packaged grains should meet the requirements of GB 7718. Labels of genetically modified food (GMF) should meet the relevant national rules and regulations.

10. Transportation and Storage

Grain should be stored in a clean and dry environment to prevent moisture, rain and pollution. There should be no mingling with poisoned, harmful, smelly substances or substances with high moisture.

The tools and containers for transportation should meet certain hygiene requirements. They should prevent from raining and pollution during the transportation.

Annex A

(Normative)

Determination of Zearalenone-Thin Layer Chromatography (TLC)

A.1 Scope

This standard defines the zearalenone determined by TLC method.

This standard is applicable to the determination of zearalenone in grains.

The detection limit of this standard is 0.03 μg.

A.2 Principle

After being extracted, cleansed, concentrated, thin-layer chromatography of silica-gel G, zearalenone in the sample will allow the visualization of blue-fluorescence spots under 254 nm UV-light. Determine the amount of zearalenone according to the fluorescence amount on the thin layer, compared with the standard.

A.3 Reagents

Unless otherwise noted, only analytical reagents, distilled water or water with a certain pure level are applied in analysis.

A 3.1 Absolute ethyl alcohol

A 3.2 Ethyl acetate and cool

A 3.3 Trichloromethane

A 3.4 1 mol /L sodium hydroxide

A 3.5 Phosphoric acid

A 3.6 Acetone

A 3.7 Silica-gel G

A 3.8 Anhydrous sodium sulfate

A 3.9 Zearalenone standard solution

Preparation of zearalenone standard solution: Dissolve 3 mg zearalenone in 100 ml absolute ethyl alcohol to make standard solution of 0.03 g/L zearalenone. Extract 1 ml standard solution and dilute it with absolute ethyl alcohol to 10 ml to make

standard solution of 3 μg/ml zearalenone. Reserve this solution in a refrigerator of 4ºC.

A 4 Apparatus

A 4.1 Micromill

A 4.2 Electric agitator

A 4.3 Ultraviolet lamp

A 4.4 Glass plate: 5 cm × 20 cm

A 4.5 Thin layer spreader

A 4.6 Microsyringe

A 5 Analysis procedure

A 5.1 Extracting and purifying

Weigh 20 g sample in powder and put the powder into 250 ml flask with stopper. Add 6 ml water and 100 ml ethyl acetate. Vibrate the flask for 1 hour. Filter the solution with folded filter paper. Weigh 25 ml filtrate into 75 ml evaporating dish and place the dish on a container with hot water to make it concentrate into dryness. Dissolve the residue with 25 ml trichloromethane in three times and transfer the solution into separating funnel of 100ml. Add 10 ml 1 mol/l sodium hydroxide solution in the previous evaporating dish, drop 1 mol/l sodium hydroxide solution along the funnel wall 1cm-2cm from the surface of trichloromethane layer, and rotate five times slightly incase emulsification. Still the funnel until the solution is separated into layers. Transfer the trichloromethane layer into second separating funnel of 100ml and add 10 ml 1 mol/l sodium hydroxide solution slowly, rotate five times slightly, remove trichloromethane layer, blend the sodium hydroxide solution in second separating funnel into the first funnel and cleanse the second funnel with few distilled water. Then transfer the cleansing liquid into the first funnel, add 5 ml trichloromethane, rotate and vibrate slightly, remove trichloromethane layer, add 5 ml trichloromethane and rotate for layer separation again, remove trichloromethane layer. Add 6ml 1.33 mol/l phosphoric acid into sodium hydroxide solution in first

funnel, neutralize the PH value of sodium hydroxide solution to 9.5 with 0.67 mol/l phosphoric acid, add 15 ml trichloromethane, rotate 20-30 times, filter the trichloromethane layer through quantitative filter paper at a slow speed with 5 g anhydrous sodium sulfate into 75 ml evaporating dish. Cleanse the funnel with few trichloromethane and blend cleansing liquid into evaporating dish. Evaporate the dish on a container with hot water into dryness, put the dish on a container with icy water and dissolve the residue with 1 ml acetone. Transfer the solution with dropper to flask with stopper as sample solution for TLC spotting.

A 5.2 Thin-layer chromatography (TLC)

A 5.2.1 Preparation of thin layer plate: Grind 3 g silica gel G with 7–8 ml distilled water to thick slurry. Move the slurry into a thin layer spreader and spread the slurry on three glass plates of 5 cm × 20 cm. Still the plates at room temperature into dryness and activate it by heating in an oven for 60 minutes at 105°C. Reserve the plates in the dryer for next steps.

A 5.2.2 Developing agent: Choose 15 ml mixture of chloroform and methanol (95:5) or 15 ml mixture of toluene, acetic acid, formic acid (6:3:1) as developing agent.

A 5.2.3 Spotting: Drop three spots of sample liquid on the baseline 2.5 cm up from bottom edge of plate with 10 µg microsyringe: 1 spot of 10 µl zearalenone standard solution, 1 spot of 30 µl of extraction solution for sample and 1 spot of 30 µl of extraction solution for sample and 10 µl zearalenone standard solution. When dropping, a cold air blower can be used to speed up the drying of plate. Drop a new spot after the previous spot is dried.

A 5.2.4 Developing: Add developing agent into a developing tank and dip the plate in agent. Remove the plate from the tank and dry when it is developed to 10 cm.

A 5.2.5 Observation and determination: Observe the plate under UVC 254 nm. If there are no blue green fluorescence spots on the spot of sample solution at the same location of spot of standard solution, the amount of zearalenone in sample determined at the method of sensibility is below 50 µg/kg; if the intensity of fluorescence spots in spot of sample solution is the same with the minimum

detectable quantity of fluorescence intensity in spot of standard solution and the locations of two fluorescence spots overlap, the amount of zearalenone in sample determined at the method of sensibility equals to 50 μg/kg; if the intensity of fluorescence spots in spot of sample solution is higher than the minimum detectable quantity of fluorescence intensity in spot of standard solution, based on the fluorescence intensity, reduce the amount of solution or add different amount of sample solution after being diluted, until the intensity of fluorescence spots in spot of sample solution is the same with the one of minimum detectable quantity.

A 6 Calculations of results

Calculate the amount of zearalenone according to the following formula (A.1):

$$x = 0.03 \times \frac{V_1}{V_2} \times \mathrm{D} \times \frac{100}{m} \quad \text{..... (A.1)}$$

Where,

x: amount of zearalenone, in the unit of μg/kg;

0.03: minimum detectable quantity of zearalenone, in the unit of μg;

V_1: volume of acetone, in the unit of ml;

V_2: volume of sample solution to drop when minimum quantity of fluorescence spots appears, in the unit of ml;

D: total dilution ratio of sample solution;

m: weight of sample when residue of acetone solution is added, in the unit of g.

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