China, Peoples Republic of

FAIRS Product Specific

FINAL GB2715-2005 Hygienic Standard for Grains

2005

Approved by:
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Report Highlights:
This is an unofficial translation of “GB2715-2005 Hygienic Standard for Grains,” which is the final version China's mandatory hygienic standard for cereals, beans, and potatoes. It entered into force Oct 1, 2005, and replaced “GB2715-1981”, which had been in force since 1982. The new standard contains broader coverage and stricter requirements than the standard it replaced, including the tolerances for pesticide residues, chemicals, mycotoxin, and toxic elements. FAS published the unofficial translation of the draft version of this new standard on Dec 13, 2004, as GAIN report CH4068. China notified this draft standard to the World Trade Organization (WTO) on Feb 11, 2004, as WTO document G/SPS/N/CHN/52.
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Executive Summary

This is an unofficial translation of "GB2715-2005 Hygienic Standard for Grains," which is the final version China's mandatory hygienic standard for cereals, beans, and potatoes. It entered into force Oct 1, 2005, and replaced “GB2715-1981”, which had been in force since 1982. The new standard contains broader coverage and stricter requirements than the standard it replaced, including the tolerances for pesticide residues, chemicals, mycotoxin, and toxic elements. FAS published the unofficial translation of the draft version of this new standard on Dec 13, 2004, as GAIN report CH4068. China notified this draft standard to the World Trade Organization (WTO) on Feb 11, 2004, as WTO document G/SPS/N/CHN/52.

In the event of any errors or omissions in this translation, the original Chinese version shall prevail. Traders should be advised that final import approval of any product is subject to the importing country's rules and regulations as interpreted by border officials at the time of product entry.
Hygienic Standard for Grains

Begin Translation

GB
National Standard of the People’s Republic of China
GB2715-2005
Replaces GB 2715-1981

Hygienic Standard for Grains

Issued on 2005-01-25
Implemented on 2005-10-01

Issued by the Ministry of Health of the People’s Republic of China and the Standardization Administration of China
Preamble

All contents of this standard are mandatory. Compared with GB2715-1981 Hygienic Standard for Grains, this standard has made the following amendments:

--Added content in scope of application as “This standard applies to raw grain and products of grain, including cereals, beans and potatoes, which are for human consumption, but does not apply to raw material used for processing edible oil.”
--Added hygienic requirements of packaging, labeling, transportation, and storage.
--Added indices of heat damaged kernel and moldy grain.
--Added indices on maximum levels of ergot, lolium temulentum, seeds of datura and other toxic plants.
--Added indices of maximum levels of De-oxynivalenol, Zearalenone, and Ochratoxin A.
--Added indices of maximum levels of Methyl Bromide, Malathion, Chlorpyrifos Methyl, Pirimiphos Methyl, Deltamethrin, and Lindane. Changed index of gross arsenic to inorganic arsenic. Eliminated indices of Cyanide and Carbon Dioxide.

This standard will be implemented on October 1, 2005 with a grace period of one year. The products produced before October 1, 2005 in accordance with relevant standards are allowed for sale until September 30, 2006.

Appendix A of this standard is a standardized one.

This standard is proposed and administered by the Ministry of Health.

This standard is drafted by Jiangsu Center for Disease Prevention and Control, Health Supervision Center of the Ministry of Health, Standardization and Quality Center of the State Administration of Grain, Institute of Nutrition and Food Hygiene of the China Center for Disease Prevention and Control, Grain Inspection Center of the Ministry of Agriculture, Exit-Entry Inspection and Quarantine Bureau in Shanghai, Exit-Entry Inspection and Quarantine Bureau in Liaoning.

People involved in the drafting of this standard are: Yuan Baojun, Zheng Yunyan, Xie Huamin, Li Xiaohui, Hou Tianliang, Guan Yuliang, Zhang Ying, Wang Xuqing.

The release history of the standard versions substituted by this standard:
Hygienic Standard for Grains

1. Scope
   This standard specifies the hygienic index for grains, testing methods, and hygienic
   requirements for packaging, labeling, transportation, and storage of grains.
   This standard applies to raw grain and products of grain, including cereals, beans and
   potato, which are for human consumption, but does not apply to raw material used for
   processing edible oil.

2. Standardized documents referenced
   The clauses in the following documents have been quoted and become provisions of
   this standard. For those quoted documents with dates, their modifications (not including
   corrections on printing errors) and revised versions do not apply to this standard.
   However, parties having reached the agreement based on this standard are encouraged
   to study whether the latest versions of the documents are applicable. For quoted
   documents without dates, their latest versions apply to this standard.

   GB 2760       Hygienic Standard for Uses of Food Additives
   GB 2763       Maximum Limits for Pesticides in Foods
   GB/T5009.11   Determination of Total Arsenic and Inorganic Arsenic in Foods
   GB/T5009.12   Determination of Lead in Foods
   GB/T5009.15   Determination of Cadmium in Foods
   GB/T5009.17   Determination of Total Mercury and Organic-Mercury in Foods
   GB/T5009.19   Determination of HCH and DDT Residues in Foods
   GB/T5009.20   Determination of Organophosphorus Pesticide Residues in Foods
   GB/T5009.22   Determination of Aflatoxin B in Foods
   GB/T5009.36   Method for Analysis of Hygienic Standard of Grains
   GB/T5009.96   Determination of Ochratoxin A in Cereals and Soybeans
   GB/T5009.110  Determination of Cypermethrin, fenvalerate, and Deltamethrin in Foods
                 of Plant Origin
   GB/T5009.111  Determination of De-oxynivalenol in Cereals and Their Products
   GB/T5009.145  Determination of Organophosphorus and Carbamic Acid Ester Pesticide
                 Residues in Foods of Plant Origin
   GB/T5494      Inspection of Grains and Oilseeds, Measures of Determination for
                 Foreign Matter and Unsound Kernels
   GB 7718       General Standard of Labeling for Pre-Packaged Food
   GB 13122      General Hygiene Regulation for Flour Mills
   SN 0649       Determination of Methyl Bromide Residue in Export Grains
   SN/T0800.7    Determination of Broken Kernels in Export Grain and Feed

3. Terminology and definition
   The following terminologies and definitions apply to this standard:

   3.1 Heat damaged kernel
       Seed kernels whose normal color has been altered due to heat generated from
       microorganism or other reasons

   3.2 Ergot
       Sclerotium formed from fungus that parasite in the ovary of grass family plant

   3.3 Lolium Temulentum
       Fruit of herbs of Grass family

   3.4 Moldy kernel
       Grains with obvious molds that hurt embryo and endosperm (or cotyledon), having no
       value for food consumption

4. Index Requirements
4.1 Sensory requirements
Must possess the natural color and odor of grains and comply with the requirements in Table 1.

<table>
<thead>
<tr>
<th>Item</th>
<th>Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat damaged kernel, (%) Wheat</td>
<td>0.5</td>
</tr>
<tr>
<td>Moldy Grain (%)</td>
<td>2.0</td>
</tr>
</tbody>
</table>

4.2 Maximum level of toxic, harmful fungi or plant seeds
Must comply with the requirements in Table 2.

<table>
<thead>
<tr>
<th>Item</th>
<th>Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ergot (%)</td>
<td></td>
</tr>
<tr>
<td>- Rice(milled), Corn, Beans</td>
<td></td>
</tr>
<tr>
<td>- Wheat, Barley</td>
<td></td>
</tr>
<tr>
<td>Must not be detected</td>
<td></td>
</tr>
<tr>
<td>0.01</td>
<td></td>
</tr>
<tr>
<td>Loliem temulentum (kernel/kg) Wheat, Barley</td>
<td>1</td>
</tr>
<tr>
<td>Seeds of datura and other toxic plants (kernel/kg) Beans</td>
<td>1</td>
</tr>
</tbody>
</table>

4.3 Physical and chemical requirements
4.3.1 Maximum level of fungi and toxins
Must comply with the requirements in Table 3.

<table>
<thead>
<tr>
<th>Item</th>
<th>Maximum level (µg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aflatoxin B</td>
<td></td>
</tr>
<tr>
<td>- Corn</td>
<td>= 20</td>
</tr>
<tr>
<td>- Rice (milled)</td>
<td>= 10</td>
</tr>
<tr>
<td>- Other</td>
<td>= 5</td>
</tr>
<tr>
<td>De-oxynivalenol (DON)</td>
<td></td>
</tr>
<tr>
<td>- Wheat, Barley, Corn, and their products of grain</td>
<td>= 1000</td>
</tr>
<tr>
<td>Zearalenone</td>
<td></td>
</tr>
<tr>
<td>- Wheat and Corn</td>
<td>= 60</td>
</tr>
<tr>
<td>Ochratoxin A</td>
<td></td>
</tr>
<tr>
<td>- Cereals, Beans</td>
<td>= 5</td>
</tr>
</tbody>
</table>

4.3.2 Index on maximum level of pollutant
Must comply with the requirements in Table 4

<table>
<thead>
<tr>
<th>Item</th>
<th>Maximum level (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lead (Pb)</td>
<td>= 0.2</td>
</tr>
<tr>
<td>Cadmium (Cd)</td>
<td></td>
</tr>
<tr>
<td>- Rice (including milled), Beans</td>
<td>= 0.2</td>
</tr>
<tr>
<td>- Wheat (including wheat flour), Corn and others</td>
<td>= 0.1</td>
</tr>
<tr>
<td>Mercury (Hg)</td>
<td>= 0.02</td>
</tr>
</tbody>
</table>
Inorganic Arsenic (As)
- Rice (milled) = 0.15
- Wheat flour = 0.1
- Other = 0.2

4.3.3 Maximum levels of pesticide residue
Maximum levels of pesticide residue must comply with table 5

Table 5 Maximum level of pesticide residue

<table>
<thead>
<tr>
<th>Item</th>
<th>Maximum Residue Limit (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phosphide (PH₃)</td>
<td>= 0.05</td>
</tr>
<tr>
<td>Methyl Bromide</td>
<td>= 5</td>
</tr>
<tr>
<td>Malathion</td>
<td>= 0.1</td>
</tr>
<tr>
<td>Chlorpyrifos Methyl</td>
<td>= 5</td>
</tr>
<tr>
<td>Pirimiphos Methyl</td>
<td>= 5</td>
</tr>
<tr>
<td>- Wheat, Rice</td>
<td>= 5</td>
</tr>
<tr>
<td>Deltamethrin</td>
<td>= 0.5</td>
</tr>
<tr>
<td>HCH</td>
<td>= 0.05</td>
</tr>
<tr>
<td>Lindane</td>
<td>= 0.05</td>
</tr>
<tr>
<td>- Wheat</td>
<td>= 0.05</td>
</tr>
<tr>
<td>DDT</td>
<td>= 0.05</td>
</tr>
<tr>
<td>Chloropicrin (measured in raw grain)</td>
<td>= 2</td>
</tr>
<tr>
<td>Heptachlor</td>
<td>= 0.02</td>
</tr>
<tr>
<td>Aldrin</td>
<td>= 0.02</td>
</tr>
<tr>
<td>Dieldrin</td>
<td>= 0.02</td>
</tr>
<tr>
<td>Other pesticides</td>
<td>Must comply with GB2763</td>
</tr>
</tbody>
</table>

5 Food Additives
5.1 Food additive quality must comply with relevant standards and regulations
5.2 Varieties and dosage of food additives must comply with GB2760

6 Testing methodology
6.1 Sensory testing
Determined in accordance with the methods prescribed in GB/T5009.36

6.2 Heat damaged kernel
Determined in accordance with the methods prescribed in SN/T0800.7

6.3 Moldy grain
Determined in accordance with the methods prescribed in GB5494

6.4 Ergot, lolium temulentum, seeds of datura and other toxic plants
Determined in accordance with the methods prescribed in GB/T5009.36

6.5 Aflatoxin B.
Determined in accordance with the methods prescribed in GB/T5009.22

6.6 De-oxynivalenol
Determined in accordance with the methods prescribed in GB/T 5009.111
6.7 Zearalenone  
Determined in accordance with the methods prescribed in Appendix A

6.8 Ochratoxin A  
Determined in accordance with the methods prescribed in GB/T5009.96

6.9 Inorganic Arsenic  
Determined in accordance with the methods prescribed in GB/T5009.11

6.10 Lead  
Determined in accordance with the methods prescribed in GB/T5009.12

6.11 Cadmium  
Determined in accordance with the methods prescribed in GB/T5009.15

6.12 Mercury  
Determined in accordance with the methods prescribed in GB/T5009.17

6.13 Phosphide, Heptachlor, Aldrin, Dieldrin, and Chloropicrin  
Determined in accordance with the methods prescribed in GB/T5009.36

6.14 Methyl Bromide  
Determined in accordance with the methods prescribed in SN/T0649

6.15 Malathion  
Determined in accordance with the methods prescribed in GB/T5009.20

6.16 Chlorpyrifos Methyl and Pirimiphos Methyl  
Determined in accordance with the methods prescribed in GB/T5009.145

6.17 Deltamethrin  
Determined in accordance with the methods prescribed in GB/T5009.110

6.18 HCH, DDT, and Lindane  
Determined in accordance with the methods prescribed in GB/T5009.19

7 Processing products of grain  
Must comply with stipulations of GB13122.

8 Packaging  
Packaging of grain should use packing materials or containers that comply with hygienic requirements, and packaging should be complete, intact, and contamination-free.

9 Labeling  
Labeling of pre-packaged grain should comply with GB7718. Genetically modified grain should be implemented in accordance with relevant government regulations.

10 Storage and transportation  
Grain storage conditions should be clean, dry, rain (moisture)-proof, and contamination-free. Must not be stored with goods (substances) that are toxic, harmful, of peculiar odor, or of high water content.  
Tools or containers used in transportation should meet hygienic requirements. Rain damage or pollutant should be avoided during transportation.
Determination of Zearalenone with Thin Layer Chromatography (TLC)

A.1 Scope

This standard prescribes the TLC method for the measurement of zearalenone in grains. This can be applied for the measurement of zearalenone. In this standard, the lowest limitation for detection is 0.03 g.

A.2 Principles

After extraction, purification, concentration and separation on the TLC made of silica gel G; zearalenone contained in the sample can emit blue fluorescence under 254 nm ultraviolet light. Thus quantification measurement can be conducted by comparing the fluorescence produced on TLC by the sample against the standard.

A.3 Reagents

If not specially designated, all reagents used in this experiment shall be of analytical purity; and the water shall be distilled water or water of reasonable purity.

A.3.1 anhydrous alcohol
A.3.2 ethyl acetate
A.3.3 chloroform
A.3.4 1 mol/L sodium hydroxide
A.3.5 phosphorous acid
A.3.6 acetone
A.3.7 silica gel G
A.3.8 anhydrous sodium sulfate
A.3.9 standard solution of zearalenone
A.3.9 preparation of standard solution

Accurately weigh 3 mg of zearalenone, add anhydrous alcohol to dissolve it and transfer the solution to a 100 ml volumetric flask, add more anhydrous alcohol to the mark. A solution thus obtained shall have a concentration of about 0.03 g/L. Pipette 1 ml of the standard solution, dilute it to 10 ml with anhydrous alcohol. There is about 3 µg of zearalenone in one ml of the standard solution. Maintained it in 4°C refrigerator for later use.

A.4 Equipment and Apparatus

A.4.1 small-scale pulverizer
A.4.2 electrical oscillator
A.4.3 ultraviolet light
A.4.4 glass plates: 5×20 cm
A.4.5 spreading device
A.4.6 micro-syringe

A.5 Analytical procedures

A.5.1 extraction and purification

Weigh 20 g of crushed sample, place it into a 250 ml bottle with stopper, add 6 ml of water and 100 ml of ethyl acetate, shake on an oscillator for 1 h, filter with folded fast filter paper, take 25 ml of filtrate, and place it in a 75 ml evaporating dish, put on water bath for concentration until dry, dissolve the residue 3 times with 25 ml chloroform, and transfer the mixture into a 100 ml separating funnel, add 10 ml 1 mol/L sodium hydroxide to the evaporating dish, and then dip sodium hydroxide solution into the separating funnel, using pipette to add 1 mol/L.
and let the liquid flow down the wall 1-2cm above the chloroform level, gently rotate 5 times the separating funnel to avoid emulsion, wait until it has been separated. Transfer the chloroform layer into a second 100ml separating funnel, then slowly add another 10ml of 1mol/l sodium hydroxide, rotated gently for 5 times, discard the chloroform layer and move the remaining sodium hydroxide solution into the former separating funnel, rinse the second funnel with small amount of distilled water, and decant the liquid into the first funnel. Add 5ml of chloroform into the first separating funnel, handshake slightly, discard the chloroform, add 5ml new chloroform and handshake again to make it separate, discard the chloroform layer, add 6ml 1.33mol/L phosphorous acid into the sodium hydroxide solution, and then adjust the pH value to 9.5 with 0.67mol/L phosphorous acid, add 15ml of chloroform into the separating funnel, handshake 20-30 times, decant the chloroform layer through a quantitative slow flow filter paper containing 5g of anhydrous sodium sulfate, and filter it into a 75ml evaporating dish, rinse the filter with small amount of chloroform, and add the chloroform into evaporating dish too, put the evaporating dish on water-bath until dry through ventilation. Accurately add 1ml of acetone after the dish has been cooled down and placed on an ice bath, mix thoroughly, and then transfer the solution with a pipette into a small bottle with stopper for TLC analytical use.

A.5.2 TLC
A.5.2.1 The preparation of TLC plates: weigh 3g of silica gel G, add 7-8ml of distilled water, blend thoroughly until the mixture becomes slurry, immediately place the slurry in the barrel of a spreading device, spread the slurry and make 3 TLC plates of 5×20cm for each, dry them at room temperature and place them in an oven for activation for about 1 h at 105? , take out and keep them in a desiccator for future use.

A.4.2.3 Developing solvent: Developing solvent can be chosen from the following combinations: chloroform-methanol (95:5) 15ml, or methylbenzene-acetic acid-methyl acid (6:3:1) 15ml.

A.5.2.3 Sample application: 3 spots of test solution are applied on the baseline 2.5 cm above the bottom end of the plate with 10µl micro-syringe: 10µl standard solution, 30µl test solution mixed with 10µl standard solution, hair drier can be used to blow cold air during the application, one drop should become dried before another one can be applied.

A.5.2.4 Spreading: Pour the spreading solvent in the developing tank, dip the plate into the solvent, spread for 10cm, take out and wave it dry.
A.5.2.5 Observation and evaluation: The plate shall be observed under an ultraviolet light (254nm). If there aren’t any cyan florescence spots beside the spots for standard solution where the sample spots should have appeared, then the zearalenone content in the sample is bellow the limitation of detection (50µg/kg); if the intensity of florescence spots of test solution equals to that of the standard solution (the limitation of detection), and that spots overlap with the spots of internal standard, then the zearalenone content in the sample is equal to the limitation of detection (50µg/kg); if the florescence produced by the spots of test solution show higher intensity than the limitation of detection, then according to the intensity of florescence, the volume of application has to be reduced or different volumes of the solution have to be applied after dilution, so that the intensity of florescence of test solution equals to that of the limitation of detection.

A.6 Result Calculation

\[ X = 0.03 \times \frac{v_1}{v_2} \times D \times 100/m \]

Here:

- \( X \): content of zearalenone, µg/kg
0.03: the limitation of detection of zearalenone, µg
V1: volume of acetone, mL
V2: volume of test solution applied to obtain intensity of florescence equals to that of the limitation of detection, mL
D: dilution factor
m: equivalent sample quantity when acetone is added to dissolve the residue, g

End of translation