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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 23200.113-2026

Replacing GB 23200.113-2018

National food safety standard - Determination of 242 pesticides and metabolites residues in foods of plant origin - Gas chromatography-tandem mass spectrometry method

食品安全国家标准 植物源性食品中242种农药及其代谢物残留量的测定 气相色谱-质谱联用法

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Ministry of Agriculture and Rural Affairs of the People's Republic of China;
State Administration for Market Regulation

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1 Scope

This document specifies the gas chromatography-tandem mass spectrometry method for the determination of the residues of 242 pesticides and their metabolites, listed in Annex A, in foods of plant origin.

It applies to the determination of the residues of those 242 pesticides and metabolites in foods of plant origin.

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 2763-2026 National food safety standard - Maximum residue limits for pesticides in food · GB/T 6682 Water for analytical laboratory use - Specification and test methods

3 Principle

The test sample is extracted with acetonitrile. The extract is cleaned up by solid-phase extraction or dispersive solid-phase extraction; samples of vegetable oil are cleaned up by gel permeation chromatography. Determination is by gas chromatography-tandem mass spectrometry, and quantification is by the internal standard method or the external standard method.

4 Reagents and materials

Unless otherwise stated, only reagents of analytical grade are used, and the water is grade one water as specified in GB/T 6682.

4.1 Reagents. Acetonitrile; ethyl acetate, chromatographic grade; toluene; cyclohexane, chromatographic grade; sodium chloride; sodium acetate; acetic acid; anhydrous magnesium sulfate; sodium citrate dihydrate; disodium citrate sesquihydrate.

4.2 Preparation of solutions. Acetonitrile-acetic acid solution (99+1): 10 mL of acetic acid made up with 990 mL of acetonitrile and mixed. Acetonitrile-toluene solution (3+1): 100 mL of toluene made up with 300 mL of acetonitrile and mixed. Gel permeation chromatography mobile phase, cyclohexane-ethyl acetate solution (1+1): 500 mL of cyclohexane made up with 500 mL of ethyl acetate and mixed.

4.3 Standard substances. The internal standard heptachlor exo-epoxide B and the standard substances of the 242 pesticides and metabolites, listed in Annex B, of purity not less than 95 %.

4.4 Preparation of standard solutions. Standard stock solutions at 1 000 mg/L, prepared by dissolving about 10 mg of each pesticide standard, and stored away from light at -18 degrees Celsius or below for up to one year. Mixed intermediate standard solutions at 20 mg/L to 50 mg/L and mixed working standard solutions at 5 mg/L, stored under the same conditions for six months and one month respectively. The internal standard solution is prepared from about 10 mg of heptachlor exo-epoxide B and diluted to 5 mg/L.

4.5 Materials. Ethylenediamine-N-propylsilanised silica (PSA), 40 micrometres to 60 micrometres; octadecylsilane bonded silica (C18), 40 micrometres to 60 micrometres; graphitised carbon black (GCB), 40 micrometres to 120 micrometres; ceramic homogenisers; microporous membrane filters, organic phase, 13 mm by 0.22 micrometres; solid-phase extraction cartridges, graphitised carbon black-amino composite, 500 mg/500 mg, 6 mL.

5 Apparatus

5.1 Gas chromatograph-triple quadrupole mass spectrometer, fitted with an electron impact (EI) source. 5.2 Gel permeation chromatograph or apparatus, with a 25 mm internal diameter by 500 mm clean-up column packed with Bio-Beads SX-3 or equivalent. 5.3 Analytical balance, readable to 0.1 mg and to 0.01 g. 5.4 High-speed homogeniser, not less than 15 000 r/min. 5.5 Centrifuge, not less than 4 200 r/min. 5.6 Tissue crusher. 5.7 Rotary evaporator. 5.8 Nitrogen evaporator with temperature control. 5.9 Vortex mixer.

6 Preparation of test samples

6.1 Preparation. The portion of the sample to be analysed is taken in accordance with Annex A of GB 2763-2026, and the sample quantity in accordance with the relevant standards or provisions. Vegetables, fruit, edible fungi and sugar crops are chopped, mixed thoroughly and either quartered or homogenised directly in the tissue crusher, then placed in polyethylene bottles or bags. Dried vegetables, fruit and edible fungi are ground and mixed. Cereals are ground so that all of the material passes a 425 micrometre standard sieve. Oilseeds and nuts are ground and mixed; tea and spices are ground and mixed; vegetable oils are simply mixed.

6.2 Storage. The portions for testing and for reserve are stored separately, at -18 degrees Celsius or below.

7 Analytical procedure

7.1 QuEChERS sample preparation, with separate procedures for vegetables, fruit, edible fungi and sugar crops; for cereals, oilseeds and nuts; and for tea and spices.

7.2 Solid-phase extraction sample preparation, comprising extraction and clean-up on a graphitised carbon black-amino cartridge.

7.5 Measurement of the sample solution. The matrix-matched working standard solution and the sample solution are injected in turn into the gas chromatograph-mass spectrometer. Identification is by retention time and ion abundance ratio, and the peak area of the quantifier ion is measured. The response of the pesticide in the sample solution shall fall within the linear range of the instrument; where it exceeds that range the extract or the sample solution is diluted appropriately and the analysis repeated.

7.6 Parallel test. A parallel determination is carried out on the same sample by the steps above.

7.7 Blank test. The same procedure is carried out in parallel without the test sample.

8 Calculation of results

The residue of each pesticide in the sample is expressed as a mass fraction in milligrams per kilogram and calculated by formula (1), (2) or (3), according to whether the internal standard method or the external standard method is used and whether a matrix-matched calibration curve is applied.

The result is the arithmetic mean of two independent determinations obtained under repeatability conditions, given to two significant figures; where the content exceeds 1 mg/kg it is given to three significant figures.

9 Precision

Under repeatability conditions, the absolute difference between two independent test results shall not exceed the repeatability limit r given in Annex E.

Under reproducibility conditions, the absolute difference between two independent test results shall not exceed the reproducibility limit R given in Annex E.

10 Other

The limits of quantification of the method of this document range from 0.002 mg/kg to 0.05 mg/kg for the various compounds, as given in Annex A.