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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 23200.121-2026

Replacing GB 23200.121-2021

National food safety standard - Determination of 352 pesticides and metabolites residues in foods of plant origin - Liquid chromatography-tandem mass spectrometry method

食品安全国家标准 植物源性食品中352种农药及其代谢物残留量的测定 液相色谱-质谱联用法

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Ministry of Agriculture and Rural Affairs of the People's Republic of China;
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Table of Contents

1 Scope

This document specifies a liquid chromatography-tandem mass spectrometry method for determining the residues of 352 pesticides and their metabolites (see Annex A) in foods of plant origin.

It applies to the determination of the residues of those 352 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 dispersive solid-phase extraction, the determination is made by liquid chromatography-tandem mass spectrometry and the quantification is by 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.

Reagents: acetonitrile (CAS 75-05-8), acetonitrile of chromatographic grade, methanol of chromatographic grade (CAS 67-56-1), sodium chloride (CAS 7647-14-5), sodium acetate (CAS 127-09-3), acetic acid (CAS 64-19-7), anhydrous magnesium sulfate (CAS 7487-88-9), trisodium citrate dihydrate (CAS 6132-04-3), disodium citrate sesquihydrate (CAS 6132-05-4), formic acid of chromatographic grade (CAS 64-18-6) and ammonium formate (CAS 540-69-2).

Solutions: acetonitrile-acetic acid solution (99+1), obtained by adding 10 mL of acetic acid to 990 mL of acetonitrile; aqueous formic acid solution (0.01 %), obtained by adding 0.1 mL of formic acid to 1 000 mL of water; methanolic formic acid solution (0.01 %), obtained by adding 0.1 mL of formic acid to 1 000 mL of methanol; aqueous ammonium formate-formic acid solution (2 mmol/L), obtained by dissolving 0.126 1 g of ammonium formate in the 0.01 % aqueous formic acid solution and diluting to 1 000 mL; and methanolic ammonium formate-formic acid solution (2 mmol/L), prepared in the same way with the 0.01 % methanolic formic acid solution.

Standards: reference standards of the 352 pesticides and metabolites listed in Annex B, of purity not lower than 95 %.

Standard solutions: stock standard solutions at 1 000 mg/L, prepared by weighing about 10 mg of each standard to the nearest 0.1 mg, dissolving it in methanol or acetonitrile according to its solubility and making up to 10 mL; they are kept in the dark at -18 degrees Celsius or below and are valid for one year. Mixed intermediate standard solutions at 20 mg/L to 50 mg/L are valid for six months and the mixed working standard solution at 5 mg/L for one month, both under the same storage conditions.

Materials: ethylenediamine-N-propyl silanised silica gel (PSA) of particle size 40 micrometres to 60 micrometres; octadecylsilane bonded silica gel (C18) of the same particle size; graphitised carbon black (GCB) of particle size 40 micrometres to 120 micrometres; ceramic homogenisers of 2 cm length and 1 cm outside diameter, or equivalent; and organic-phase microporous membrane filters of 13 mm by 0.22

micrometres, or equivalent.

5 Apparatus

Liquid chromatograph-triple quadrupole mass spectrometer fitted with an electrospray ionisation (ESI) source.

Analytical balance readable to 0.1 mg and to 0.01 g.

Centrifuge with a speed of not less than 4 200 r/min.

Tissue homogeniser and vortex mixer.

6 Preparation of test samples

The part of the sample to be tested is that specified in Annex A of GB 2763-2026, and the sampling quantity follows the relevant standards or rules.

Vegetables, fruits, edible fungi and sugar crops are cut up and mixed thoroughly, then either quartered or homogenised directly in the tissue homogeniser, and placed in polyethylene bottles or bags. Dried vegetables, fruits and edible fungi are ground and mixed thoroughly. Cereals are ground until they pass entirely through a 425 micrometre standard sieve or an equivalent one. Oil crops and nuts, and tea and condiments (spices), are ground and mixed thoroughly. Vegetable oils are simply mixed and placed in polyethylene bottles.

Test samples are stored separately for testing and for reserve, at -18 degrees Celsius or below.

7 Analytical procedure

Vegetables, fruits, edible fungi and sugar crops: weigh 10 g of the test sample to the nearest 0.01 g into a 50 mL plastic centrifuge tube, add 10 mL of acetonitrile and one ceramic homogeniser, shake vigorously for 1 min, add 4 g of anhydrous magnesium sulfate, 1 g of sodium chloride, 1 g of trisodium citrate dihydrate and 0.5 g of disodium citrate sesquihydrate, shake vigorously for 1 min and centrifuge at 4 200 r/min for 5 min. Transfer a measured volume of the supernatant to a plastic centrifuge tube containing the drying agent and the clean-up material (150 mg of anhydrous magnesium sulfate and 25 mg of PSA per millilitre of extract); for darkly coloured samples add also 2.5 mg of GCB per millilitre. Vortex for 1 min, centrifuge at 4 200 r/min for 5 min and filter the supernatant through the membrane.

Cereals, oil crops and nuts: weigh 5 g of the test sample, add 10 mL of water, vortex and let stand for 30 min, then add 15 mL of the acetonitrile-acetic acid solution and one ceramic homogeniser, shake for 1 min, add 6 g of anhydrous magnesium sulfate and 1.5 g of sodium acetate, shake for 1 min and centrifuge. Clean up with 150 mg of anhydrous magnesium sulfate, 50 mg of C18 and 50 mg of PSA per millilitre of extract.

Tea and condiments (spices): weigh 2 g of the test sample and proceed as for cereals, cleaning up with 150 mg of anhydrous magnesium sulfate, 50 mg of C18, 50 mg of PSA and 25 mg of GCB per millilitre of extract.

Vegetable oils: weigh 2 g of the test sample, add 5 mL of water and 10 mL of acetonitrile with one ceramic homogeniser, shake for 1 min, add 4 g of anhydrous magnesium sulfate, 1 g of sodium chloride, 1 g of trisodium citrate dihydrate and 0.5 g of disodium citrate sesquihydrate, shake and centrifuge, then clean up with 150 mg of anhydrous magnesium sulfate, 50 mg of C18 and 50 mg of PSA per millilitre of extract.

Note: when sulfonylurea herbicides, cyclohexenone herbicides, sulfonamide herbicides, fluazinam, spirotetramat-enol, spirotetramat-enol-glucoside, bentazone, 8-hydroxybentazone, thidiazuron, the cyazofamid metabolite CCIM, probenazole or isoxaflutole-diketonitrile are to be determined, the amount of PSA is reduced to 5 mg per millilitre of extract for vegetables, fruits, edible fungi, sugar crops and vegetable oils, and to 10 mg per millilitre for cereals, oil crops and nuts.

Liquid chromatographic conditions: C18 column of 2.1 mm internal diameter by 100 mm, particle size 1.8 micrometres or equivalent; mobile phase A the aqueous ammonium formate-formic acid solution and mobile phase B the methanolic one, run on the gradient of Table 1; flow rate 0.3 mL/min; column temperature 40 degrees Celsius; injection volume 2 microlitres.

Mass spectrometric conditions: electrospray ionisation source; simultaneous positive and negative ion scanning; spray voltage 5 500 V in positive mode and -4 500 V in negative mode; source temperature 350 degrees Celsius; nebuliser gas and auxiliary heating gas both at 0.345 MPa; multiple reaction monitoring with at least two ion pairs selected for each pesticide, all ions detected in time segments in order of elution. The retention times, precursor ions, product ions and ion-pair parameters of each pesticide are given in Annex C.

Matrix-matched calibration curve: blank samples of the same or a similar nature to the samples under test are prepared as in 7.1 to 7.4 to give a blank matrix solution, which is used to dilute the mixed working standard solution to 0.002 mg/L, 0.005 mg/L, 0.01 mg/L, 0.02 mg/L, 0.05 mg/L, 0.1 mg/L, 0.2 mg/L and 0.5 mg/L. Not fewer than five concentration points are chosen according to the performance of the instrument, and the curve is plotted with the peak area of the quantifier ion against the concentration.

Identification: the relative error between the retention time of the target peak in the sample and that of the corresponding standard shall be within plus or minus 2.5 %. When the retention times agree, all the selected product ions of the target compound are present, and the ion abundance ratio in the sample does not deviate from that of a matrix standard solution of comparable concentration by more than the values of Table 2 (plus or minus 20 % where the ratio is greater than 50, plus or minus 25 % where it is greater than 20 up to