Chinastandards.org

CCS G 25

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

chinastandards.org - PREVIEW

GB 23200.112-2018

National food safety standard -- Determination of 9 carbamate pesticides and their metabolite residues in plant-derived foods -- Liquid chromatography -- Post-column derivatization

Issued on: June 21, 2018 **Implemented on: December 21, 2018**

Issued by: National Health Commission of the People's Republic of China, State Administration for Market Regulation

Preview: page 1 of 5 shown, full document available. Complete English PDF, instant download:
https://chinastandards.org/standards/gb-23200-112-2018

Table of Contents

chinastandards.org · PREVIEW

1 Scope

This standard specifies a liquid chromatography-post-column derivatization method for the determination of residues of nine carbamate pesticides and their metabolites (see Appendix A) in plant-derived foods: This standard is applicable to the determination of residues of 9 carbamate pesticides and their metabolites in plant-derived foods:

2 Normative reference documents

The following documents are essential for the application of this document: For dated references, only the dated version applies to this document:

For undated referenced documents, the latest version (including all amendments) applies to this document:

3 principles

The sample is extracted with acetonitrile, and the extract is purified by solid-phase extraction or dispersive solid-phase extraction: It is detected by a high-performance liquid chromatograph with a fluorescence detector and a post-column derivatization system, and quantified by an external standard method:

4 Reagents and materials

Unless otherwise stated, only analytically pure reagents are used in the analysis, and the water is first-grade water specified in GB/T 6682:

4:1 Reagents 4:1:1 Acetonitrile (CH3CN, CAS number: 75-05-8):

4:1:2 Methanol (CH3OH, CAS number: 67-56-1): chromatographically pure:

4:1:3 Dichloromethane (CH2Cl2, CAS number: 75-09-2): chromatographically pure:

Preview: page 2 of 5 shown, full document available. Complete English PDF, instant download:
https://chinastandards.org/standards/gb-23200-112-2018

4:1:4 Toluene (C1Hs, CAS number: 108-88-3): chromatographically pure:

4:1:5 Sodium chloride (NaCl, CAS number: 7647-14-5):

4:1:6 Phthalaldehyde (C8H8O2, CAS number: 643-79-8):

4:1:72-Dimethylaminoethanethiol hydrochloride (C4H2CINS, CAS number: 13242-44-9), or equivalent:

4:1:8 Anhydrous magnesium sulfate (MgSO CAS number: 7487-88-9):

4:1:9 Sodium acetate (CH3COONa, CAS number: 6131-90-4):

4:1:10 Sodium hydroxide (NaOH, CAS number: 1310-73-2):

4:1:11 Sodium tetraborate decahydrate (Na2B4O2.10H2O, CAS number: 1303-96-4):

4:2 Solution preparation 4:2:1 Methanol-dichloromethane solution (1 99, volume ratio): Measure 10 mL of methanol and add it to 990 mL of dichloromethane, and mix well:

4:2:2 Acetonitrile-toluene solution (3 1, volume ratio): Measure 100 mL of toluene and add it to 300 mL of acetonitrile, and mix well:

4:2:3 Sodium hydroxide solution (0:05 mol/L): Weigh 2:0g of sodium hydroxide, dissolve it in water and adjust the volume to 1000mL, and mix well:

4:2:4 Sodium tetraborate decahydrate solution (4g/L): Weigh 4:0g of sodium tetraborate decahydrate, dissolve in water and dilute to 1000mL, and mix well:

4:2:5 OPA reagent: Weigh 50:0 mg o-phthalaldehyde, dissolve in 5 mL methanol, and mix; then weigh 1:0 g 2-dimethylaminoethanethiol hydrochloride, dissolve in 5 mL sodium tetraborate decahydrate solution (4:2:4), mix well; pour the above two solutions into 490mL of sodium tetraborate decahydrate solution Liquid (4:2:4), mix well:

4:3 Standard products Please refer to Appendix A for the standards of 9 carbamate pesticides and their metabolites, with a purity of >=95%:

4:4 Preparation of standard solution 4:4:1 Standard stock solution (1000mg/L): Accurately weigh 10mg (accurate to 0:1mg) of each pesticide standard, dissolve it in methanol and dilute to 10mL: The standard stock solution should be stored at -18°C away from light and is valid for 1 year:

4:4:2 Mix standard solution: Accurately draw a certain amount of individual pesticide stock solution into a 10mL volumetric flask, and dilute to the mark with methanol: Mix the standard solution and store it away from light at 0 degrees C~4 degrees C: The validity period is 1 month:

4:5 Materials 4:5:1 Solid phase extraction column 1: amino packing (NH2) 500mg, 6mL:

4:5:2 Solid phase extraction column 2: graphitized carbon black packing (GCB) 500mg,

chinastandards.org . PREVIEW

Preview: page 3 of 5 shown, full document available. Complete English PDF, instant download:
https://chinastandards.org/standards/gb-23200-112-2018

amino packing (NH2) 500mg, 6mL:

4:5:3 Ethylenediamine-N-propylsilane silica gel (PSA): 40 um ~ 60 um:

4:5:4 Octadecylsilane modified silica gel (Cs): 40 um ~ 60m:

4:5:5 Ceramic homogeneous proton: 2cm (length) x 1cm (outer diameter):

4:5:6 Microporous filter membrane (organic phase): 0:22umx25mm:

5 Instruments and Equipment

5:1 Liquid chromatograph: equipped with post-column derivatization reaction device and fluorescence detector (FLD):

5:2 Analytical balance: sensitive to 0:1mg and 0:01g:

5:3 High-speed homogenizer: the rotation speed is not less than 15000r/min:

5:4 High-speed centrifuge: the rotation speed is not less than 4200 r/min:

5:5 Tissue masher:

5:6 Rotary evaporator:

5:7 Nitrogen blower: temperature controllable:

5:8 Vortex oscillator:

6 Preparation of specimens

6:1 Sample preparation Accurately pipette 8mL of the supernatant into a 15mL plastic centrifuge tube containing 1200mg anhydrous magnesium sulfate, 400mg PSA and 400mg Ci: Vortex and mix for 1 minute, then centrifuge at 4200 r/min for 5 minutes: Pipette 5mL of the supernatant into a 10mL centrifuge tube: , evaporate to almost dryness by blowing nitrogen in a 50°C water bath, add 2:00mL of methanol accurately, vortex to mix, filter with a microporous membrane (4:5:6), and wait for testing:

7:1:5 Vegetable oil Weigh 3g of sample (accurate to 0:01g) into a 50mL plastic centrifuge tube, add 5mL of water, 15mL of acetonitrile, and add 6g of anhydrous Magnesium sulfate, 1:5g sodium acetate and 1 ceramic homogeneous proton, shake vigorously for 1 min, centrifuge at 4200 r/min for 5 min:

Accurately pipette 8mL of the supernatant into a 15mL plastic centrifuge tube containing 1200mg anhydrous magnesium sulfate, 400mg PSA and 400mg Cis: Vortex and mix for 1 minute, then centrifuge at 4200 r/min for 5 minutes: Pipette 5mL of the supernatant into a 10mL centrifuge tube: , evaporate to almost dryness by blowing nitrogen in a 50°C water bath, add 1:00mL of methanol accurately, vortex to mix, filter with a microporous membrane (4:5:6), and wait for testing:

chinastandards.org · PREVIEW

Preview: page 4 of 5 shown, full document available. Complete English PDF, instant download:
https://chinastandards.org/standards/gb-23200-112-2018

7:2 Determination 7:2:1 Instrument reference conditions a) Chromatographic column: C8, 250mmx4:6mm (inner diameter), 5um (particle size);

b) Column temperature: 42 degrees C;

c) Fluorescence detector: lambdaex=330 nm, lambdaem=465 nm;

d) Mobile phase and gradient elution conditions, see Table 1;

e) Post-column derivatization: 0:05 mol/L sodium hydroxide solution, flow rate 0:3mL/min; OPA reagent, flow rate 0:3mL/min; hydrolysis temperature, 100°C; derivatization temperature, room temperature;

f) Injection volume: 10uL:

7:2:2 Standard working curve Accurately draw a certain amount of mixed standard solution, and dilute it with methanol step by step to a mass concentration of 0:01mg/L, 0:05mg/L, 0:1mg/ L:0:5mg/L and 1:0mg/L standard working solutions for liquid chromatography determination: Draw a standard curve with the pesticide mass concentration as the abscissa and the peak area of the chromatographic peak as the ordinate:

7:2:3 Qualitative and quantitative 7:2:3:1 Qualitative Qualitative based on the retention time of the target pesticide: The retention time of the target pesticide chromatographic peak in the test sample is compared with the retention time of the corresponding standard chromatographic peak, and the difference should be within +/-0:05 min: Positive samples need to be replaced with a Cis column for qualitative confirmation:

7:2:3:2 Quantification Quantification by external standard method:

7:3 Determination of sample solution Inject the mixed standard working solution and sample solution into the liquid chromatograph in sequence, qualitatively measure the retention time, and measure the target pesticide chromatographic peak surface Product, according to formula (1), the content of each pesticide component is obtained: The response value of the pesticide in the sample liquid to be tested should be within the linear range of the quantitative determination of the instrument: When it exceeds the linear range, appropriate multiple dilutions should be made based on the measured concentration before analysis:

7:4 Parallel tests Carry out parallel tests on the same sample according to the provisions of 7:1 to 7:3:

7:5 Air self-test Except that no sample is added, parallel operations shall be carried out according to the provisions of 7:1 to 7:4:

chinastandards.org · PREVIEW

Preview: page 5 of 5 shown, full document available. Complete English PDF, instant download:
https://chinastandards.org/standards/gb-23200-112-2018