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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 39076-2020

Textiles - Determination of neonicotinoid pesticide residues

纺织品 新烟碱类农药残留的测定

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1 Scope

China's national method for determining neonicotinoid pesticide residues in textiles. It specifies the principles, the reagents and materials, the apparatus and the analytical procedure. Neonicotinoids are the most widely used insecticide class in the world, applied to cotton among many other crops, and they are systemic - taken up into the plant rather than sitting on its surface - so residues can persist into the fibre rather than being washed off. They are also the compounds at the centre of the pollinator decline debate and are restricted or banned in agriculture across several jurisdictions. Their presence in a finished textile matters on two counts: as a consumer exposure, since a garment is worn against skin for hours, and as a trade matter, since the restricted substance lists that govern textile exports have begun to include them. The determination is by liquid chromatography with mass spectrometry, at concentrations of a few milligrams per kilogram.

This standard specifies the method of use of liquid chromatography-tandem mass spectrometry (LC-MS/MS) for the determination of 7 neonicotinoid pesticide residues in textiles. This standard applies to all types of textile products.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this standard.

GB/T 6682 Water for analytical laboratory use - Specification and test methods

3 Principles

The specimen is ultrasonically extracted by acetonitrile; the extract is filtered through a filter membrane, then measured and confirmed by liquid chromatography-tandem mass spectrometry (LC-MS/MS), quantified by external standard method.

4 Reagents and materials

4.1 Acetonitrile: Chromatographically pure.

4.2 The 7 standard neonicotinoid pesticides: purity >= 98%, as shown in Appendix A.

4.3 Standard stock solution (approximately 1000 mg/L): Respectively accurately weigh appropriate amounts of neonicotinoid pesticide standard products (accurate to 0.0001 g). Use acetonitrile (4.1) to prepare each substance to the standard stock solution which has a mass concentration of approximately 1000 mg/L.

Note: When stored at 0 °C ~ 4 °C in the dark, the validity period of the standard stock solution is 12 months.

4.4 Mixed standard working solution (approximately 10 mg/L): Accurately pipette an appropriate amount of standard stock solution (4.3). Use acetonitrile (4.1) to prepare a mixed standard working solution which has a concentration of approximately 10 mg/L. Then, prepare the standard working solution of other concentrations as needed.

Note: When stored at 0 °C ~ 4 °C in the dark, the validity period of the mixed standard working solution is 3 months.

4.5 Water: Class II water that meets the requirements of GB/T 6682.

5 Apparatus and equipment

5.1 Liquid chromatography-tandem mass spectrometer (LC-MS/MS): It is equipped with electrospray ionization source (ESI).

5.2 Ultrasonic generator: The working frequency is 40 kHz.

5.3 Balance: Sensitivity is 0.0001 g and

0.01 g, respectively.

5.4 Conical flask: It has a ground stopper; the capacity is 100 mL.

5.5 Polytetrafluoroethylene filter membrane: The pore size is 0.22 μm.

6 Analytical procedures

6.1 Extraction Take a representative sample. Cut it into small pieces below 5 mm x 5 mm.

Mix well. Weigh

1.0 g (accurate to

0.01

g) and mix the specimen uniformly. Place it in a conical flask (5.4). Accurately add 20 mL of acetonitrile (4.1). Close the stopper. Place the conical flask in the ultrasonic generator (5.2). Extract it at room temperature for 60 minutes. Use polytetrafluoroethylene filter membrane (5.5) to filter the sample solution into a sample bottle, which is used for LC- MS/MS analysis. Where: Xi - The content of neonicotinoid pesticide i in the specimen, in milligrams per kilogram (mg/kg); ci - The concentration of neonicotinoid pesticide i in the sample as calculated from the standard curve, the unit is milligrams per liter (mg/L); V - Volume of sample liquid, the unit is milliliters (mL); F - Dilution factor; m - The mass of the specimen, in grams (g).

7.2 Results presentation The test results are represented by the test results of various neonicotinoid pesticides. The calculation results are kept to one decimal place. When it is below the lower limit of determination, the test result is "not detected".

8 Low limit of determination and precision

8.1 Lower limit of determination The lower limit of determination is

0.2 mg/kg.

8.2 Precision In the same laboratory, when the same operator uses the same equipment, according to the same test method, to carry out tests of same tested object independently in a short time, at the 95% confidence level, the absolute difference of the two independent test results obtained is not more than 20% of the arithmetic mean of the two measured values.

9 Test report

The test report shall give at least the following contents:

a) Sample description;

b) Number of this standard;