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# NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA



# GB/T 44165.6-2024

## Determination of key chemicals in consumer products - Part 6: Acrylamide

消费品中重点化学物质检测方法 第6部分:丙烯酰胺

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**Standardization Administration of the PRC**

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## 0 Place in the series

This is the sixth part of GB/T 44165, the series that supplies test methods for the chemicals dealt with in GB/T 39498. Six of the nine planned parts have been published, the remaining three covering polychlorinated naphthalenes, perfluorooctane sulfonate together with perfluorooctanoic acid, and hexabromocyclododecane.

The document opens with the same warning as the other parts: those using it should have practical experience of regular laboratory work, it does not point out every safety problem that may arise, and the user is responsible for taking suitable safety and health measures and for meeting national regulations. Clause 3 states that the part needs no terms or definitions of its own.

## 4 Principle

For migration, the test piece is spiked with a carbon-13 labelled acrylamide internal standard and the migration test is run with water as the simulant, the liquid then being filtered through a membrane. For total content, the test portion is spiked with the same internal standard, diluted and filtered through a membrane.

In both cases the prepared liquid goes to a liquid chromatograph coupled to a tandem mass spectrometer working in multiple reaction monitoring, and quantification is by internal

standard.

## 5 Reagents and materials

Unless stated otherwise the reagents are of analytical grade and the water is grade one water as recommended in GB/T 6682. Methanol and formic acid are of chromatographic grade.

The acrylamide reference material is of 98 per cent purity, CAS number 79-06-1; the labelled internal standard is acrylamide-13C3, CAS number 287399-26-2.

Both stock solutions are made up in methanol at 100 mg per litre and kept in a freezer at minus 20 degrees Celsius, where they last three months. The internal standard working solution is made by diluting 1.0 mL of the internal standard stock to the mark with methanol to give 10 mg per litre, and is prepared fresh.

The calibration series is made by transferring the stock solution to a 10 mL flask, adding 0.1 mL of the internal standard working solution and diluting with water to give acrylamide at 2.5, 5, 10, 20, 50 and 100 µg per litre, the internal standard standing at 100 µg per litre throughout. These too are prepared fresh.



## 6 Apparatus

A liquid chromatograph with a tandem mass spectrometer fitted with a triple quadrupole detector; an electronic analytical balance reading to 0.0001 g; a horizontal shaker running at not less than 60 revolutions per minute; an ultrasonic cleaning bath; a vortex mixer; a centrifuge running at not less than 10000 revolutions per minute; and polyethersulfone microporous membrane filters of 0.22 µm.

## 7 Samples and test portions

For the migration test the piece is cut with a suitable tool to a surface area of 10 +/- 1 square centimetres. Where the test part is thicker than 1 mm the thickness is counted in. The edges of the test part are to look smooth. Where the laboratory sample has a surface area under 10 square centimetres it is tested whole, without cutting. Test pieces are kept frozen at minus 20 degrees Celsius.

For the migration itself the piece is placed in the extraction bottle with tweezers, 100 mL of water and 0.1 mL of the internal standard stock solution are added, the bottle is sealed and shaken horizontally at room temperature at 60 +/- 5 revolutions per minute for 60 +/- 5 min. A portion of the migration liquid is then filtered through the membrane and measured.

For total content the sample is mixed even and 1.0 g, weighed to 0.0001 g, goes into a centrifuge tube with 8 mL of water and 0.1 mL of the internal standard working solution. The tube is vortexed for 1 min, sonicated for 60 min, cooled to room temperature, spun at 10000

revolutions per minute for 10 min, and the supernatant is transferred to a 10 mL flask and made up to the mark with water. A portion of that extract is filtered through the membrane and measured.

## 8 Test procedure

Because the result depends on the instrument used, no general set of parameters can be given for the liquid chromatograph and tandem mass spectrometer; the following set has been shown to work. The column is a C18 of 2.1 mm by 100 mm with 1.5 µm particles, or an equivalent. The mobile phase is methanol and water carrying 0.1 per cent formic acid by volume, in the ratio 90 to 10, at 0.2 mL per minute, with the column at 40 degrees Celsius and an injection volume of 5 µL. Ionisation is by positive electrospray, acquisition is in multiple reaction monitoring, the cone voltage is 40 V and the collision energy 12 eV. Acrylamide is followed from a precursor at 72 m/z to products at 44 m/z and 55 m/z, the latter serving for quantification; the labelled internal standard is followed from a precursor at 75 m/z to products at 43 m/z and 58 m/z, the latter serving for quantification.

For identification, the retention time of the peak found in the sample is to match that of the reference material, all the selected ions are to be present in the background subtracted mass spectrum of the sample, and the relative abundance ratio of those ions is to match that of a reference material of comparable concentration within the tolerance of Table 1. That tolerance widens as the abundance falls: +/- 20 per cent above a relative abundance of 50, +/- 25 per cent between 20 and 50, +/- 30 per cent between 10 and 20, and +/- 50 per cent at 10 or below. The reagent blank is injected in the same way and is to show nothing that interferes with the analyte.

For quantification the calibration series is injected under the same conditions and a curve is drawn with the concentration of the calibration solution on the horizontal axis and the ratio of the acrylamide peak area to that of the labelled internal standard on the vertical; the linear correlation coefficient is to exceed 0.99. A blank test is run through the whole of the sample preparation and measurement, the test portion alone being left out.

## 9 Data processing and method limits

The migration is obtained from the difference between the mass concentration read off the curve for the sample solution and that read off for the blank, multiplied by the dilution factor, and is given in micrograms per litre. The total content is obtained from the mass concentration read off for the sample solution, the make-up volume and the dilution factor, divided by the mass of the test portion, and is given in micrograms per kilogram. Both are reported to three significant figures.

For migration the detection limit of the method is 1.0 µg per litre and the quantification limit 2.5 µg per litre. For content the detection limit is 10 µg per kilogram and the quantification

limit 25 µg per kilogram.

## 11 Precision and report

Where two independent results are obtained in the same laboratory, by the same operator on the same equipment, by the same method and within a short time, on the same test object, the absolute difference between them is not to exceed 10 per cent of their arithmetic mean.

The report gives at least a description of the test sample, the number of this document, the test result, any departure from the analytical procedure laid down, anything unusual noticed during the test, and the date of the test.

An informative annex reproduces the total ion chromatograms and the multiple reaction monitoring traces of the acrylamide reference material and of the labelled internal standard.