ICS 03.120.99
CCS A 00

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 44165.5-2024

Determination of key chemicals in consumer products - Part 5: Phenol

消费品中重点化学物质检测方法 第5部分:苯酚

Issued on: June 29, 2024 **Implemented on: June 29, 2024**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

Table of Contents

0 Warning and position in the series

The document opens with a warning that the people using it are to have practical experience of regular laboratory work, that it does not point out every possible safety problem, and that the user is responsible for taking suitable safety and health measures and for complying with the conditions laid down in national regulations.

The foreword records that this is part 5 of GB/T 44165, Determination of key chemicals in consumer products, and that parts 1 to 6 had been published: short-chain chlorinated paraffins, styrene migration, chloroethanes, 1,4-dichlorobenzene, phenol and acrylamide. The introduction states that the series provides methods for the substances covered by GB/T 39498, the guide to the control of key chemicals in consumer products, that it is intended as a uniform or supplementary method standard where none exists, and that it is planned to run to nine parts, the last three being polychlorinated naphthalenes, perfluorooctane sulfonate and perfluorooctanoic acid, and hexabromocyclododecane.

3 Terms and definitions

3.1 flexible modeling materials: water-soluble gel-type or soft mouldable materials designed to be kneaded, stretched or shaped.

4 Principle

4 states the principle. For the phenol content the sample is extracted with methanol under ultrasound and filtered through a membrane before measurement; for the phenol migration water serves as the migration medium, the phenol is extracted from the specimen and the liquid filtered through a membrane before measurement. The solution is then measured on a high performance liquid chromatograph and quantified by the external standard method.

5 Reagents and materials

5 requires all reagents to be of analytical grade unless otherwise stated: methanol of chromatographic grade; water of grade 1 according to GB/T 6682; phenol reference material of 98 % purity, CAS number 108-95-2.

5.4 prepares the stock standard solution by weighing a suitable amount of the phenol reference material into a volumetric flask and dissolving and making up to the mark with methanol, giving 1000 mg/L.

5.5 and 5.6 prepare the series of working standard solutions by diluting the stock solution, with methanol for the content determination and with water for the migration determination, to mass concentrations of 0.5 mg/L, 1.0 mg/L, 2.0 mg/L, 5.0 mg/L, 10.0 mg/L and 20 mg/L.

5.7 and 5.8 call for organic and aqueous filter membranes with a pore size of 0.45 micrometre.

6 Apparatus

6 lists the apparatus: a high performance liquid chromatograph fitted with a diode array detector; a gas chromatograph-mass spectrometer; an electronic analytical balance accurate to 0.1 mg; a vortex mixer; an ultrasonic extractor of 250 W and 53 kHz or equivalent; a centrifuge running at not less than 10000 r/min; an extraction bottle, that is a flat-bottomed conical flask of 250 mL with a stopper; a fully automatic temperature-controlled shaker whose temperature can be held to +/-2 °C; stoppered glass bottles of 40 mL; stoppered colorimetric tubes of 10 mL; stoppered centrifuge tubes; and stainless steel tweezers.

7 Sample pre-treatment

7.1.1 treats a solid sample: it is cut with a suitable tool into particles no larger than 5 mm by

5 mm by 5 mm, 1.0 g is weighed accurately to 0.0001 g into a 40 mL stoppered glass bottle, 10.0 mL of methanol is added accurately, the bottle is placed in the ultrasonic extractor and sonicated at room temperature for 60 min +/-2 min, and after cooling to room temperature the extract is filtered through the organic membrane.

7.1.2 treats a liquid sample or a flexible modelling material: 1.0 g is weighed accurately to 0.0001 g into a 10 mL stoppered colorimetric tube, 8 mL of methanol is added, the tube is vortex mixed and sonicated for 15 min +/-2 min, and after cooling to room temperature methanol is added to the mark and the tube mixed. Where necessary the liquid is transferred to a 15 mL stoppered plastic centrifuge tube and centrifuged at 10000 r/min for 10 min, and the supernatant is filtered through the organic membrane.

7.2.1 governs the taking of migration specimens. A specimen of 10 cm2 +/-1 cm2 is cut from a representative test sample with a suitable tool, in a circular, square, rectangular or other shape that reduces the cut edges, the sampling parameters being given in Annex A, and a thin part of the sample is chosen. Where the specimen is thicker than 1 mm the surface area of the thickness is to be included in the calculation of the surface area. The surface is to be smooth and loose particles are to be removed. A test sample with a surface area smaller than 10 cm2 need not be cut and the whole sample serves as the specimen; such a specimen is tested with 100 mL of water. Where the specimen is thin and tends to stick to the wall of the extraction bottle, a small hole may be pierced in it and a small metal or other suitable object passed through the hole and fixed there to hold it away from the wall. Where the way the specimen is cut would have a marked effect on the migration, the whole uncut sample or sample part is used as the specimen and the migration run with a proportionate volume of water, 10 mL for each square centimetre of surface.

7.2.2 governs the migration itself: the specimen is placed in the extraction bottle with stainless steel tweezers, 100 mL of water at 37 °C +/-2 °C is added, the bottle is sealed and placed in the temperature-controlled shaker and shaken horizontally at 37 °C +/-2 °C and 60 r/min +/-5 r/min for 60 min +/-5 min. A suitable amount of the migration liquid is then filtered through the aqueous membrane.

7.3 requires a blank test to be run for both the content and the migration determination following the same steps but without the specimen.

8 Determination

8.1 states that the result depends on the instrument used and that no general chromatographic parameters can be given, then lists parameters that have been shown to work: a C18 column of 250 mm by 4.6 mm with a particle size of 5 micrometre or equivalent; a mobile phase of methanol and water in the proportion 60 to 40, isocratic; a flow rate of 1.0 mL/min; a column temperature of 40 °C; an injection volume of 10 microlitre; and a detection wavelength of 271 nm.

8.2 identifies the analyte by comparing the ultraviolet spectrum and the retention time of the peak with those of the standard, the retention time reference being Annex B; where the retention time, the spectrum and the reference material agree, the sample is judged to contain phenol. Confirmation by gas chromatography-mass spectrometry is allowed where needed, with the parameters of Annex C.

8.3 quantifies by injecting the series of working standard solutions under the conditions of 8.1, plotting the peak area against the concentration and using the external standard method.

9 Calculation of results

9.1 gives formula (1) for the phenol content. The printed equation is broken by the extraction and is not reproduced here; its legend reads: the phenol content of the specimen in milligrams per kilogram; the mass concentration of phenol in the sample solution and in the blank solution, both in milligrams per litre; the volume to which the solution was made up, in millilitres; the dilution factor; and the mass of sample taken, in grams. The result is kept to three significant figures.

9.2 gives formula (2) for the phenol migration, the mass concentration of phenol in the migration liquid being read directly from the calibration curve. Its legend reads: the phenol migration from the specimen in milligrams per litre; the mass concentration of phenol in the migration liquid of the specimen and in the blank migration liquid, both in milligrams per litre; and the dilution factor. The result is again kept to three significant figures.

10 Limit of quantification of the method

10 fixes the limit of quantification at 5 mg/kg for the phenol content and at 0.5 mg/L for the phenol migration.

12 Test report

12 requires the test report to give at least a description of the specimen, the number of this document, the test result, any departure from the analytical procedure laid down, any abnormal phenomenon observed during the test, and the date of the test.

A Annex A (informative) Examples of sampling of polymer materials in the phenol migration test

Table A.1 gives reference sampling parameters for specimens less than 1 mm thick, in three columns, cut shape, size and specific surface area in square centimetres, and three rows that reconcile: a circle of radius 1.79 cm giving 10.1; a square of side 3.18 cm giving 10.1; and a rectangle 5 cm long and 2 cm wide giving 10.0.