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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 31604.47-2023

**National food safety standard - Food contact materials and
products - Determination of fluorescent substances in paper,
cardboard and paper products**

食品安全国家标准 食品接触材料及制品 纸、纸板及纸制品
中荧光性物质的测定

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National food safety standard - Food contact materials and products - Determination of fluorescent substances in paper, cardboard and paper products

1 Scope

This standard specifies the method for the determination of fluorescent substances in food contact paper, cardboard, and paper products.

This standard is applicable to the determination of fluorescent substances in food contact paper, cardboard, and paper products.

2 Principle

Whether the sample contains fluorescent substances is determined by directly observing under UV light whether there is an obvious fluorescence phenomenon (that is, obvious blue or purple fluorescence) in food contact paper, cardboard, and paper product samples. If there are many discontinuous small spots of fluorescence in the sample or the fluorescence phenomenon is not obvious, it is extracted with an alkaline extracting solution, then the extract is acidified, and gauze is used to absorb the fluorescent substances in the extract; under irradiation with the ultraviolet light, the gauze is observed whether there is obvious fluorescence phenomenon to confirm whether the sample contains fluorescent substances.

3 Reagents and materials

Unless otherwise stated, all reagents used are of analytical grade and the water is first-grade water specified in GB/T 6682. All reagents and materials used shall have no fluorescence under UV light.

3.1 Reagents

3.1.1 Acetonitrile (C_2H_3N): chromatographically pure.

3.1.2 Triethylamine ($C_6H_{15}N$).

3.1.3 Sodium hydroxide (NaOH): guaranteed reagent.

3.1.4 Hydrochloric acid (HCl).

sample, cut into paper scraps of approximately 5 mm×5 mm, and then crush them into cotton wool with a high-speed pulverizer for later use. If it cannot be tested immediately, it shall be placed in a clean polyethylene plastic bag and stored at room temperature away from light. For each sample, prepare 2 test portions in parallel.

Take the paper sample with no fluorescence observed as a blank sample and process it according to the above steps.

5.2 Direct measurement of fluorescent substances and judgment of results

In a darkroom or dark box, place the 100 cm² sample cut according to 5.1.1 about 20 cm below the UV light source, turn on the power switch of the UV lamp, select the detection wavelengths of 254 nm and 365 nm respectively, and directly observe whether fluorescence phenomenon appears on the sample.

For food contact paper and cardboard, at any wavelength of 254 nm or 365 nm, if the fluorescent area of any one of the five samples is greater than 5 cm², the fluorescent substance in the sample is judged to be positive, otherwise, the fluorescent substance in the sample is judged to be negative; for food contact paper products, at any wavelength of 254 nm or 365 nm, if the fluorescence area of any one of two samples from the same batch is greater than 5 cm², the fluorescent substance in the sample is judged to be positive, otherwise, the fluorescent substance in the sample is judged to be negative.

If there are multiple discontinuous small spots of fluorescence in the sample, or if the sample has fluorescence but is not obvious, it is necessary to continue the confirmation test of the fluorescent substance.

5.3 Confirmation of fluorescent substances

5.3.1 Preparation of non-fluorescent gauze

Use scissors and a right-angled triangle to cut out several pieces of gauze about 5 cm×5 cm in size. Then immerse the cut gauze in the alkaline extracting solution and soak it in a 40 °C water bath for 30 minutes. Use tweezers to take out the gauze and rinse it with water 2~3 times; after squeezing out most of the liquid, place the gauze in a dry and ventilated place to dry naturally. If the test cannot be carried out immediately, it shall be placed in a clean polyethylene plastic bag and stored at room temperature away from light.

5.3.2 Preparation of standard control gauze

Weigh 2.0 g of the uniformly crushed blank sample prepared according to 5.1.2 (accurate to 1 mg) into a 250 mL Erlenmeyer flask, add 0.5 mL of standard working solution (40.0 mg/L), then place in a dark place (the illumination is required to be less than 20 lx), add 100 mL of alkaline extracting solution, and conduct ultrasonic extraction at 50 °C for 40 minutes. After the extraction is completed, cool to room

temperature, filter the extract into a chicken heart bottle through a glass funnel containing a little glass wool, or centrifuge at 3500 r/min for 5 minutes to obtain a clear extract. Concentrate the extract under reduced pressure to 40 mL~50 mL at 50 °C, transfer the concentrated solution to a 250 mL beaker, then wash the chicken heart bottle with water, and transfer the washing liquid to the 250 mL beaker; adjust the pH to 3~5 with a hydrochloric acid solution, and add water to approximately 100 mL. Then, immerse a piece of gauze without fluorescence phenomenon in the extract and adsorb in a 40 °C water bath for 30 minutes. After taking out the gauze with tweezers, squeeze out most of the liquid, then fold the gauze into 4 layers, each layer has an area of about 2.5 cm×2.5 cm, and place it in a glass watch glass.

5.3.3 Sample extraction and adsorption (preparation of sample gauze)

Weigh 2.0 g of the uniformly crushed sample prepared according to 5.1.2 into a 250 mL Erlenmeyer flask, and perform the rest of the operations in accordance with the steps "then place in a dark place..., and place it in a glass watch glass" in 5.3.2.

5.3.4 Blank test (preparation of blank sample gauze)

Weigh 2.0 g of the blank sample that was crushed evenly according to 5.1.2, and perform the remaining operations in accordance with 5.3.3 simultaneously with the sample test.

5.3.5 Confirmatory test for fluorescent substances

In a darkroom or dark box, place the watch glass containing the standard control gauze, blank sample gauze, and two parallel sample gauze about 20 cm below the UV light source, turn on the power switch of the UV lamp, then select the detection wavelength of 254 nm and 365 nm respectively, and observe directly. At the two wavelengths of 254 nm and 365 nm, if the standard control gauze has obvious fluorescence (that is, there is obvious blue or purple fluorescence) and the blank sample gauze has no obvious fluorescence, then the sample extraction and adsorption operation steps are considered correct; if at any wavelength, the standard control gauze has no obvious fluorescence phenomenon or the blank sample gauze has fluorescence phenomenon, it is considered that the sample extraction and adsorption operation steps are improper, and the test needs to be carried out again. After confirming that the sample extraction and adsorption procedures are correct, compare the two parallel sample gauze with the blank sample gauze to observe whether the sample gauze has obvious blue or purple fluorescence.

5.3.6 Determination of confirmation test results of fluorescent substances

At the two wavelengths of 254 nm and 365 nm, if there is no obvious fluorescence phenomenon in the two parallel sample gauze (that is, no obvious blue or purple fluorescence), the fluorescent substance in the sample is judged to be negative; if two

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