GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 85.010

Y 30

GB/T 27741-2018

Replacing GB/T 27741-2011

Paper and board - Determination of migratable fluorescent whitening agents

纸和纸板 可迁移性荧光增白剂的测定

Issued on: December 28, 2018 **Implemented on: July 01, 2019**

Issued by: State Market Regulatory Administration;
Standardization Administration of PRC.

Table of Contents

Paper and board - Determination of migratable fluorescent whitening agents

Caution - Personnel using this standard shall have practical experience in formal chemistry experiment work. This standard does not address all safety issues; users are responsible for taking appropriate safety and health measures and ensuring compliance with the relevant national regulations.

1 Scope

This standard specifies qualitative and quantitative methods for the determination of migratable fluorescent whitening agents in paper and board.

The qualitative analysis method of the migratable fluorescent whitening agent in this standard is applicable to various paper and board; the UV-visible spectrophotometry in the quantitative analysis method is suitable for the mobile fluorescent whitening agent (in VBL) in paper and board. The high performance liquid chromatography is suitable for the determination of the content of seven fluorescent whitening agents (see Appendix A) in paper and board; other fluorescent whitening agents can be tested with reference to it. The migratable fluorescent whitening agent in the pulp can be tested by reference to qualitative and quantitative methods in the standard.

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 450 Paper and board - Sampling for testing and identification of machine and cross direction, wire side and felt side

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

3 Terms and definitions

The following terms and definitions apply to this document.

5.3.1 Conical flask: 250 mL.

5.3.2 Glass funnel: Glass core funnel with specification G1.

5.3.3 Glass watch glass.

5.3.4 Balance: The sensitivity is 0.1 g.

5.3.5 Constant temperature oscillating water bath: The temperature can be controlled at (40 ± 2) °C.

5.3.6 UV lamp: A device with eye protection at a wavelength of 254 nm and a wavelength of 365 nm.

5.3.7 pH meter: The reading is accurate to 0.01.

5.4 Test procedure

5.4.1 Extraction of specimen

Accurately weigh 2.0 g of the specimen and place it in a conical flask (5.3.1). Add 100 mL of the extract (5.2.4) to the flask. Whilst slowing shaking the flask, extract it at room temperature for 10 min. Then use a glass funnel (5.3.2) to filter it. Use the hydrochloric acid solution (5.2.2) to adjust the pH of filtrate to 3.0 ~ 5.0. Immerse the gauze (5.2.1) in the filtrate. Heat it in a constant temperature oscillating water bath (5.3.5) at a temperature of (40 ± 2) °C for 30 min. Use tweezers to take out the gauze. Then squeeze the filtrate dry and fold it into 4 layers. Place it on the watch glass (5.3.3).

5.4.2 Blank test

Except for not adding specimen, follow the procedures of 5.4.1 to perform the blank test.

5.4.3 Testing

Place the watch glass on which the specimen gauze (5.4.1) and the blank test gauze (5.4.2) are placed at about 20 cm under the UV lamp (5.3.6) in the dark room or dark box, to observe the gauze's fluorescence phenomenon. Each specimen is subjected two parallel determinations.

5.5 Determination of results

If the specimen gauze of two parallel tests is not significantly fluorescent compared with the blank test gauze, it is judged that the specimen has no migratable fluorescent whitening agent; if there is one blank test in the specimen gauze of two parallel tests is significantly fluorescent than the blank test gauze, perform two parallel tests again. If the re-tested specimen gauze

different working conditions of different instruments is different. The linearity of the standard solution concentration range from 1 mg/L to 20 mg/L has been proved to be satisfactory.

6.1.2.6 Microporous membrane: 0.45 μm.

6.1.3 Instruments

6.1.3.1 UV-visible spectrophotometer.

6.1.3.2 Balance: The sensitivities are 0.1 mg and 0.01 g, respectively.

6.1.3.3 Constant temperature oscillating water bath: The temperature can be controlled at (80 ± 2) °C.

6.1.3.4 Flask with stopper: 250 mL.

6.1.4 Test procedure

6.1.4.1 Extraction of specimen

Weigh about 1.00 g of the specimen in a stoppered flask (6.1.3.4). Accurately add 50 mL of the extract (6.1.2.2). Then shake it in a (80 ± 2) °C constant temperature water bath (6.1.3.3) to extract it for 60 min. Cool to room temperature in the dark. Use the microporous membrane (6.1.2.6) to filter it. Retain the filtrate. Each specimen is subjected to parallel tests.

6.1.4.2 Blank test

Except for not adding the specimen, follow the procedures of 6.1.4.1 to perform the blank test, to prepare the blank solution.

6.1.4.3 Determination

Adjust the wavelength of the UV-visible spectrophotometer to 348 nm. Determine the absorbance of the fluorescent whitening agent VBL in the standard working solution (6.1.2.5), the specimen solution (6.1.4.1), the blank solution (6.1.4.2). Use the determination results of the standard working solution to draw a standard curve. Calculate the mass concentration of the migratable fluorescent whitening agent in the specimen filtrate and the blank solution.

6.1.5 Calculation and representation of results

Calculate the content of the migratable fluorescent whitening agent w (in VBL) in the specimen according to formula (1):

$$w = \frac{(\rho - \rho_0) \times V \times 1\,000}{m \times 1\,000} \qquad \cdots\cdots(1)$$

dissolve it. Make its volume reach to 100 mL.

Note: The standard stock solution is stored in the refrigerator at 2 °C ~ 4 °C in the dark, valid for 6 months.

6.2.2.7 Fluorescent whitening agent standard working solution: Accurately transfer a certain volume of standard stock solution (6.2.2.6) as needed. Use the extract (6.2.2.5) to dilute it to the appropriate concentration of series standard working solution. The standard working solution is prepared before use. The linear range of the measurement under different working conditions of different instruments is different. The linearity of the standard solution concentration range of the seven fluorescent whitening agent from 0.1 mg/L to 10.0 mg/L has been proved to be satisfactory.

6.2.2.8 Microporous membrane: 0.45 μm.

6.2.3 Instruments

6.2.3.1 High performance liquid chromatograph, equipped with a fluorescence detector (FLD).

6.2.3.2 Balance: The sensitivities are 0.1 g and 0.1 mg.

6.2.4 Test procedure

6.2.4.1 Extraction of specimen

It is performed according to 6.1.4.1.

6.2.4.2 Blank test

Except for not adding specimen, follow the procedures of 6.1.4.1 to carry out the blank test.

6.2.4.3 Determination

6.2.4.3.1 HPLC analytical conditions

Since the test results depend on the instrument used, it is not possible to give a general parameter for chromatographic analysis. The following parameters have been proven to be suitable for testing.

a) Column: C18 column, 5.0 μm, 4.6 mm × 250 mm or equivalent;

b) Flow rate: 1.0 mL/min;

c) Column temperature: 40 °C;

d) Injection volume: 1 μL;

V - Volume of extract, in milliliters (mL);

m - The absolute dry mass of the specimen, in grams (g).

The calculation result is rounded off to one decimal place.

7 Detection limit and recovery rate

The detection limit of UV-visible spectrophotometry is 20.0 mg/kg. The detection limit of high performance liquid chromatography is 1.0 mg/kg. Add an appropriate amount of standard solution to the negative sample and analyze it according to Chapter 6. The recovery of the fluorescent whitening agent is 80% ~ 120%.

8 Precision

In the same laboratory, the same operator uses the same equipment according to the same test method to perform mutually independent tests for the same tested object within a short period of time. The absolute difference between the two independent test results obtained is not greater than 15% of the arithmetic mean of the two measured values.

9 Test report

The test report shall include at least the following:

a) The source and description of the sample;

b) The number of this standard and the method used;

c) The test results;

d) If the number of measurements is more than two, describe the number of measurements;

e) Details on any deviation from this standard;

f) Any abnormal phenomena observed during the test;

g) Test date.

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------